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L11: Entry 3 of 29 File: USPT Sep 24, 2002

DOCUMENT-IDENTIFIER: US 6455486 B1
TITLE: Anti-spotting and anti-filming hard surface cleaning formulations and methods

CLAIMS:

7. An aqueous hard surface cleaning formulation comprising a hard surface cleaning effective amount of: (a) a graft copolymer of polyalkylene oxide with vinyl ester, the graft copolymer having a molecular weight within the range of about 5,000 to 50,000; (b) at least one polycarboxylate selected from the group consisting of acrylic acid/maleic acid copolymers, having a molecular weight within the range of about 1,000 to 100,000 and polyacrylic acid having a molecular weight of from about 1,000 to about 100,000; (c) at least one nonionic surfactant selected from the group consisting of alcohol alkoxylates, alcohol block alkoxylates, and polyoxyethylene/polyoxypropylene triblock surfactants; and (d) one or more water soluble organic solvents selected from the group consisting of N-methyl pyrrolidone and ethylene glycol monobutyl ether.

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L11: Entry 28 of 29 File: DWPI Jan 13, 1998

DERWENT-ACC-NO: 1998-126376
DERWENT-WEEK: 199812


TITLE: Water dispersing coating composition with good water resistance and soil resistance - comprising ethylene glycol derivative and di:ethylene glycol derivative as film forming auxiliary agent

PATENT-ASSIGNEE: DAICEL CHEM IND LTD (DAIL)

PRIORITY-DATA: 1996JP-0169629 (June 28, 1996)

PATENT-FAMILY:

| PUB-NO | PUB-DATE | LANGUAGE | PAGES | MAIN-IPC |
|---|---|---|---|---|
| JP 10007976 A | January 13, 1998 | | 006 | C09D157/00 |

APPLICATION-DATA:

| PUB-NO | APPL-DATE | APPL-NO | DESCRIPTOR |
|---|---|---|---|
| JP 10007976A | June 28, 1996 | 1996JP-0169629 | |

INT-CL (IPC): C09 D 5/02; C09 D 125/02; C09 D 133/06; C09 D 133/14; C09 D 157/00

ABSTRACTED-PUB-NO: JP 10007976A
BASIC-ABSTRACT:

A water dispersing coating compsn. having a glass transition temp. of at least 50 deg. C is obtained by emulsion polymerizing a monomer mixture in the presence of a surfactant in an aq. medium. The monomer mixture comprises: (a) 70-99.9 wt.% of an ethylenic unsaturated monomer; and (b) 0.1-10 wt.% of a water soluble ethylenic unsaturated monomer. The coating compsn. comprises 0.5-10 wt.% of a mixture of ethylene glycol derivative and diethylene glycol derivative as a film forming auxiliary agent at a mixing ratio of the ethylene glycol derivative and the diethylene glycol derivative of 2:8-8:2 by wt.. Also claimed the above where: (1) the surfactant is at least one of a fatty acid ester of a higher alcohol, alkyl benzene sulphonate, fatty acid sulphonate and their salts, an alkyl ester, alkyl ether or alkylphenyl ether of a polyethylene glycol; (2) the surfactant is an amphoteric surfactant in which the anionic part is carboxylate, sulphonate or sulphate and the cationic part is ami ne salt, quaternary ammonium salt; (3) (a) is at least one of 1-24C alkyl (meth)acrylate, 2-8C hydroxyalkyl (meth)acrylate, styrene, vinyltoluene, alpha-methylstyrene, N-vinyl pyrrolidone, vinylpyridine, glycidyl (meth)acrylate, (meth)acrylate having 1-methyl-2-pyrrolidone, 1-ethyl-2-pyrrolidone, 1-methyl-2-oxazolidone, 1-ethyl-2-oxazolidone, 1-methyl-2-imidazolidone or 1-ethyl-2-imidazolidone; (4) (b) is at least one of (meth)acrylic acid, maleic acid, crotonic acid, polyoxyethylene (meth)methacrylate, acrylamide, N-methylol acrylamide or N-butoxymethylacrylamide; (5) the ethylene glycol derivative is at least one of butyl cellosolve, ethyl cellosolve and the diethylene glycol derivative is at least one of ethylcarbitol, butylcarbitol

USE - The compsn. is suitably used for a coating agent.

ADVANTAGE - The compsn. has excellent water resistance and soil resistivity

ABSTRACTED-PUB-NO: JP 10007976A
EQUIVALENT-ABSTRACTS:

CHOSEN-DRAWING: Dwg.0/0

DERWENT-CLASS: A13 A14 A82 E19 G02
CPI-CODES: A12-B01A; E10-A09B4; E10-A09B8; E10-E04M1; E10-E04M3; E10-G02H1; E10-G02H2; G02-A02C; G02-A02D;

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L11: Entry 26 of 29 File: EPAB Oct 12, 1988

PUB-NO: EP000286075A2
DOCUMENT-IDENTIFIER: EP 286075 A2
TITLE: Detergent composition.

PUBN-DATE: October 12, 1988

INVENTOR-INFORMATION:

| NAME | COUNTRY |
|---|---|
| HAN, SHAW-LIN | |
| LAI, KUO-YANN | |
| DULIBA, EDWARD P | |

ASSIGNEE-INFORMATION:

| NAME | COUNTRY |
|---|---|
| COLGATE PALMOLIVE CO | US |

APPL-NO: EP88105516
APPL-DATE: April 7, 1988

PRIORITY-DATA: US03663587A (April 10, 1987)

US-CL-CURRENT: 510/423; 510/496
INT-CL (IPC): C11D 3/30; C11D 3/43
EUR-CL (EPC): C11D003/00; C11D003/30, C11D003/43

ABSTRACT:

CHG DATE=19990617 STATUS=O> Liquid, caustic-free, pre-spotting compositions that remove baked-on food residues from hard surfaces at ambient temperatures are provided which comprise: a. from about 1 to 40%, preferably 4 to 20%, of a surfactant selected from the group consisting of anionic surfactants, nonionic surfactants and mixtures thereof; b. from about 1 to 10% of a builder selected from the group consisting of polyphosphates, pyrophosphates, citrates, carbonates, and mixtures thereof; c. from about 0.2% to 2% of an amine selected from the group consisting of monoethanolamine, diethanolamine, triethanolamine and mixtures thereof; d. water; and e. further comprising from about 3 to 50% of a solvent, which solvent is selected from the groups consisting of: i) sulfolane, propylene glycol monoethyl ether acetate, dipropylene glycol monomethyl ether acetate, ethylene glycol monoethyl ether acetate, diethylene glycol monoethyl ether acetate, diethylene glycol dimethyl ether, ethylene glycol dimethyl ether, diethylene glycol diethyl ether, and mixtures thereof; ii) diethylene glycol monobutyl ether, ethylene glycol monobutyl ether, and N-methyl 2-pyrrolidone and mixtures thereof; and iii) a mixture of two solvents, the first such solvent comprising 5-17% of an acetate selected from the group consisting of ethyl acetate and n-propyl acetate, and the second such solvent comprising 15-34% of a solvent selected from the group consisting of acetone, N-methyl 2-pyrrolidone and methyl ethyl ketone, wherein the ratio of the first solvent to the second solvent may range from 1:4 to 1:2. Additionally, such formulations may include: f. from about 3 to 22% of imidazole; and g. up to 6% of a foam booster, a foam stabilizer, a viscosity adjusting agent, and mixtures thereof.

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L12: Entry 22 of 90 File: USPT Apr 9, 2002

DOCUMENT-IDENTIFIER: US 6369010 B1
TITLE: Method and composition for preventing pitch deposits in paper mills using resinous mechanical pulps

Abstract Text (1):
The present invention is a method and composition for preventing the deposition of contaminants from resinous pulp and/or recycled pulp in the press section of a pulp and paper process. The composition of the invention includes a cleaning solvent comprising a blend of aromatic hydrocarbons containing between nine and eleven carbon atoms and a surfactant comprising an alcohol ethoxylate. The composition of the invention can be applied to the felts in the press felt section of a pulp and paper process to break down and loosen deposited contaminants and the contaminants can be removed thereby preventing the need for batch cleaning and downtime in the press felt section.

Brief Summary Text (4):
Paper machines that make paper from resinous thermomechanical pulps have historically suffered from operational problems in the press section of the paper process. In particular, resinous materials or pitch can become trapped within the felt structure and can deposit on the surface of press felts, rolls, and uhle boxes, thereby hindering the effective removal of water from the paper web as it is being produced. As a result, these deposits cause reduced operational efficiency and reduce the quality of the paper being produced.

Brief Summary Text (5):
Heretofore, paper mill operators have attempted to deal with the problem of pitch deposition by applying surfactant-based cleaners, solvents, or mixtures thereof to the press section. The surfactant-based cleaners and solvents are either fed continuously while the paper machine is in operation and making paper (continuous cleaning) or when the paper machine is down and not making paper (batch cleaning) to remove pitch from the press section.

Brief Summary Text (6):
One particular class of surfactants that has traditionally been used to remove pitch deposits from felts using both continuous and batch cleaning processes are the alkyl phenol ethoxylates, e.g., the nonylphenol alkoxylates. U.S. Pat. Nos. 5,167,767; 5,520,781 and 5,575,893 describe the use of ethoxylated phenols such as nonylphenol ethoxylates for use as felt conditioners. Although these surfactants are commonly used, the federal government has begun to closely regulate the alkyl phenol ethoxylates due to concerns about their biodegradation and their possible estrogenic effects on aquatic life. In particular, the federal government has listed certain nonylphenol ethoxylates and related compounds and derivatives (e.g. nonylphenol based phosphate esters, octyl phenols, dinonyl phenols, dodecyl phenols and related alkylphenol based compounds) as SARA Toxic Release Chemicals and requires extensive reporting and tracking of many of these compounds.

Brief Summary Text (8):
In addition to environmental problems, most surfactant-based cleaners and solvents typically are not effective enough to prevent the need for downtime during operation of the paper mill. Although many prior art products extend the time between downtime periods or batch cleanings, these products generally do not eliminate the need for downtime. A is readily understood by those skilled in the art, it is advantageous

for mills to operate with limited downtime. In particular, the felts in the pressing section can typically be used for between four and eight weeks before they are mechanically worn out and need to be replaced. Therefore, if a mill can run for four to eight weeks without downtime using a continuous cleaning process, it is economically advantageous.

Brief Summary Text (9):
The problem of pitch deposition on press section equipment has been further complicated by recent changes in the operation of paper mills. For example, the mill operators have begun to reuse water produced during operation of the paper mill to reduce fresh water consumption. Thus, contaminants that used to be eliminated from the mill now become more concentrated in the press section and show a greater tendency to deposit on the press section equipment.

Brief Summary Text (11):
Therefore, there is a need in the art to produce a cleaning composition that can effectively remove pitch from felts and other pressing equipment in a continuous cleaning process without requiring downtime and batch cleaning. Furthermore, there is a need to produce a cleaning composition that is environmentally safe that effectively removes inks, adhesives, and other contaminants from pressing equipment.

Brief Summary Text (14):
The present invention provides a composition that includes a cleaning solvent comprising a blend of aromatic hydrocarbons containing between nine and eleven carbon atoms and a surfactant comprising an alcohol ethoxylate. The combination of the cleaning solvent and the alcohol ethoxylate, and particularly a branched or secondary alcohol ethoxylate, produces synergistic results in removing pitch and is useful in removing other contaminants from press machinery including ink, sizing agents, coatings, waxes and stickies. The composition is also substantially free of alkyl phenols, alkylphenol ethoxylates, naphthalene and other environmentally undesirable compounds. In addition, the composition of the invention provides effective cleaning while using a minimal amount of VOC's.

Brief Summary Text (15):
In accordance with the invention, the composition for inhibiting the deposition of pitch and other deposits on pulp and paper machinery includes a cleaning solvent comprising a blend of aromatic hydrocarbons containing between nine and eleven carbon atoms and preferably having a flash point of greater than 140.degree. F., and an alcohol ethoxylate, preferably a branched or secondary alcohol ethoxylate, and more preferably a tridecyl alcohol ethoxylate. Preferably, the solvent of the composition is substantially free of naphthalene and the surfactant of the composition is substantially free of alkyl phenols and alkylphenol ethoxylates such as nonylphenol ethoxylates. The surfactant preferably includes between 6 and 16 moles ethylene oxide, more preferably between 8 and 10 moles ethylene oxide, per mole of alcohol. The mass ratio of cleaning solvent to alcohol ethoxylate in the present composition is preferably from about 1:10 to about 10:1, more preferably from about 1:3 to about 3:1. In addition to the cleaning solvent and the alcohol ethoxylate, the present composition can also include formulation solvents, water, additional surfactants, buffers and other additives.

Brief Summary Text (16):
The present invention further includes a continuous method for inhibiting the deposition of contaminants from resinous pulps and recycled pulps on machinery in a pulp pressing process. According to the present method, an advancing paper stock material is contacted with an advancing pressing felt to remove moisture from the paper stock material. After the advancing pressing felt contacts the paper stock material, a deposition-inhibiting effective amount of the cleaning composition described above is applied to the advancing felt, preferably by spraying, to loosen pitch and other deposited contaminants from the felt. The loosened contaminants are then removed from the advancing pressing felt and the advancing pressing felt is again contacted with paper stock material to remove moisture from the paper stock material. The present method operates continuously to reduce or eliminate the deposition of pitch and other contaminants thereby allowing the press section to operate without the need for downtime during the four to eight week life span of the

(19)日本国特許庁（ＪＰ）　(12)公開特許公報（A）　(11)許出願公開番号

特開平10－7976

(43)公開日　平成10年(1998)１月13日

| (51)Int.Cl.[6] | 識別記号 | 庁内整理番号 | ＦＩ | | 技術表示箇所 |
|---|---|---|---|---|---|
| Ｃ０９Ｄ 157/00 | ＰＤＣ | | Ｃ０９Ｄ 157/00 | ＰＤＣ | |
| 5/02 | ＰＰＴ | | 5/02 | ＰＰＴ | |
| 125/02 | ＰＦＢ | | 125/02 | ＰＦＢ | |
| 133/06 | ＰＧＣ | | 133/06 | ＰＧＣ | |
| 133/14 | | | 133/14 | | |

審査請求　未請求　請求項の数８　ＯＬ　（全６頁）

(21)出願番号　特願平8－169629

(22)出願日　平成８年(1996)６月28日

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(54)【発明の名称】　水分散性被覆組成物

(57)【要約】

【課題】　改善された耐水性、耐汚染性を備えた皮膜を形成する水分散性被覆組成物を提供する。

【解決手段】　界面活性剤の存在下、水系媒質中にて、樹脂量に対して70～99.9重量％のエチレン性不飽和モノマーとこのモノマーと共重合可能な 0.1～10重量％の水溶性エチレン性不飽和モノマーを含むモノマー混合物と乳化共重合せしめて得られる、樹脂量に対して 0.5～10重量％の混合重量比が２／８～８／２のエチレングリコール誘導体とジエチレングリコール誘導体の混合物を造膜助剤として含んだ、50℃以上のTg値（ポリマーのガラス転移点）を有する水分散性被覆組成物。

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【特許請求の範囲】

【請求項1】　界面活性剤の存在下、水系媒質中にてモノマー混合物と乳化共重合せしめて得られる、50℃以上のTg値（ポリマーのガラス転移点）を有する水分散性被覆組成物であって、

前記モノマー混合物が、樹脂量に対して(a) 70～99.9重量%のエチレン性不飽和モノマー、および前記(a) エチレン性不飽和モノマーと共重合可能な 0.1～10重量%の(b) 水溶性エチレン性不飽和モノマーを含み、

前記組成物が、エチレングリコール誘導体とジエチレングリコール誘導体の混合物を、造膜助剤として、樹脂量に対して 0.5～10重量%含み、および前記エチレングリコール誘導体／ジエチレングリコール誘導体の混合重量比が、2／8～8／2である、

ことを特徴とする水分散性被覆組成物。

【請求項2】　前記界面活性剤が、高級アルコールの脂肪酸エステル塩、アルキルベンゼンスルホン酸塩、脂肪族スルホン酸塩、およびこれら塩類の一種以上の組み合わせからなるグループから選択されたアニオン系界面活性剤である、請求項1に記載の水分散性被覆組成物。

【請求項3】　前記界面活性剤が、ポリエチレングリコールのアルキルエステル型、アルキルエーテル型、アルキルフェニルエーテル型、およびこれら活性剤タイプの一種以上の組み合わせからなるグループから選択されたノニオン系界面活性剤である、請求項1に記載の水分散性被覆組成物。

【請求項4】　前記界面活性剤が、アニオン部分をカルボン酸塩、スルホン酸塩、硫酸エステル塩、そしてカチオン部分をアミン塩、第4級アンモニウム塩とした両性界面活性剤である、請求項1に記載の水分散性被覆組成物。

【請求項5】　前記(a) エチレン性不飽和モノマーが、アクリル酸メチル、アクリル酸エチル、アクリル酸プロピル、アクリル酸ブチル、アクリル酸ヘキシル、アクリル酸シクロヘキシル、アクリル酸2-エチルヘキシル、アクリル酸ラウリル、アクリル酸ステアリル、メタクリル酸メチル、メタクリル酸エチル、メタクリル酸プロピル、メタクリル酸ブチル、メタクリル酸ヘキシル、メタクリル酸シクロヘキシル、メタクリル酸2-エチルヘキシル、メタクリル酸ラウリル、メタクリル酸ステアリルなどの（メタ）アクリル酸のC１～C24のアルキルまたはシクロアルキルエステル、

ヒドロキシエチルアクリレート、ヒドロキシエチルメタクリレート、ヒドロキシプロピルアクリレート、ヒドロキシプロピルメタクリレートなどの（メタ）アクリル酸のC２～C８のヒドロキシアルキルエステル、

スチレン、ビニルトルエン、α－メチルスチレン、N－ビニルピロリドン、ビニルピリジンなどの芳香族不飽和モノマー、

グリシジルアクリレート、グリシジルメタクリレートなどのエポキシ基含有の（メタ）アクリル酸エステル、

アクリル酸1-メチル2-イミダゾリドン、アクリル酸1-エチル2-イミダゾリドン、メタクリル酸1-メチル2-イミダゾリドン、メタクリル酸1-エチル2-イミダゾリドンなどの（メタ）アクリル酸のイミダゾール環含有のC１～C24のアルキルエステル、

アクリル酸1-メチル2-ピロリドン、アクリル酸1-エチル2-ピロリドン、メタクリル酸1-メチル2-ピロリドン、メタクリル酸1-エチル2-ピロリドンなどの（メタ）アクリル酸のピロール環含有のC１～C24のアルキルエステル、

アクリル酸1-メチル2-オキサゾリドン、アクリル酸1-エチル2-オキサゾリドン、メタクリル酸1-メチル2-オキサゾリドン、メタクリル酸1-エチル2-オキサゾリドンなどの（メタ）アクリル酸のオキサゾール環含有のC１～C24のアルキルエステル、および前掲した化合物の一種以上の組み合わせ、

からなるグループから選択されたモノマーである、請求項1乃至4のいずれかに記載の水分散性被覆組成物。

【請求項6】　前記(b) 水溶性エチレン性不飽和モノマーが、

アクリル酸、メタクリル酸、マレイン酸、クロトン酸、ポリオキシエチレン鎖を有する（メタ）アクリレート、アクリルアミド、N－メチロールアクリルアミド、N－ブトキシメチルアクリルアミド、および前掲した化合物の一種以上の組み合わせ、

からなるグループから選択されたモノマーである、請求項1乃至5のいずれかに記載の水分散性被覆組成物。

【請求項7】　前記エチレングリコール誘導体が、ブチルセロソルブ、エチルセルソルブ、およびこれらの組み合わせからなるグループから選択されたエチレングリコールモノアルキルエーテルである、請求項1乃至6のいずれかに記載の水分散性被覆組成物。

【請求項8】　前記ジエチレングリコール誘導体が、エチルカルビトール、ブチルカルビトールなどのジエチレングリコールモノアルキルエーテル、ジエチレングリコールジエチルエーテル、およびこれらの組み合わせからなるグループから選択されたジエチレングリコールジアルキルエーテルである、請求項1乃至7のいずれかに記載の水分散性被覆組成物。

【発明の詳細な説明】

【0001】

【発明の属する技術分野】本発明は、水分散性被覆組成物、特に、優れた耐久性と耐汚染性を備えた塗膜を形成する水分散性被覆組成物に関する。

【0002】

【従来の技術および発明が解決しようとする課題】塗料等のコーティング剤においては、環境保全および生体（人体）への安全衛生面の観点から、塗料の無公害化ないしは安全衛生化が強く要望されており、従来の溶剤型

塗料に代わって、エマルジョン塗料の用途が拡大されつつあり、関心の高まりがみられる。 それに伴い、これらエマルジョン塗料においても高度の塗膜性能が要求されるようになり、無機質基材に塗布された場合の塗膜の耐久性と耐汚染性の向上は共に重要な課題として認識されている。

【０００３】エマルジョン塗料は、エマルジョン粒子間の熱的融着を介して初めて均一な塗膜が形成されるため、ポリマーのガラス転移点（Tg値）を一層低く設定するか、あるいは多量の造膜助剤、可塑剤等を添加する必要がある。 そのため、水性エマルジョンを造膜して得られた塗膜は、溶剤系塗料による塗膜と比較して、塗膜の耐汚染性が劣るという欠点がある。

【０００４】最近、これらの問題に関して、フッ素含有モノマーを共重合した水性エマルジョン、または水性エマルジョン同士のブレンドによる塗膜性能の向上が研究されている。 この方法によれば、共重合可能なモノマーの種類が制約され、皮膜硬度、高耐久性による皮膜の汚染性、製造コストの面などの点においてまだ改良が必要とされている。

【０００５】また、少ない溶剤量で均一な塗膜を得るための方法として、Tg値の異なる二種類のポリマーエマルジョンのブレンド物を主成分とした塗料の製法がある。

この場合、溶剤量の低減、耐水性の向上は図れるものの、耐汚染性に関しては、低Tg値のポリマーが存在するため、汚染物質の除去効率が悪く、さらにはポリマー同士の相溶性という問題が残されている。

【０００６】また、コア／シェル型エマルジョンでは、内層に高Tg値のポリマー組成を有するエマルジョン粒子を、また外層に低Tg値のエマルジョン粒子を配する構成となっており、これによりエマルジョン粒子の造膜性の改善と、各粒子間の融着に関する、高Tg値のポリマー組成物主導の耐汚染性の改善をも目指している。 しかしながら、これら両種のエマルジョンを用いて得られる塗膜では、内層と外層とのTg値の差を大きくするに従い、耐汚染性は相応に改善されるものの、内外両層のポリマー同士の相溶性の不良に基づいた塗膜の耐久性の劣化という問題点がある。 内層と外層とのTg値差が小さい場合、ポリマーの相溶性は良好となるので、この方法は塗膜の耐久性を向上させる目的においては、有効な手段と考えられるが、塗膜の耐汚染性は逆に劣化することから、耐汚染性の改善は今後の問題として残されている。

【０００７】このように、有機ポリマーを用いた研究では、耐汚染性等の改善において限界が認められ、そのため、水ガラスやコロイダルシリカといった無機ポリマーと有機ポリマーとの併用による耐久性および耐汚染性の改良研究もまた、数多くなされている。

【０００８】実際のところ、水ガラスまたはコロイダルシリカと有機ポリマー・エマルジョンとのブレンド物を主成分とする塗料から得られる塗膜は、硬度も高く、かつ耐汚染性も良好であるが、無機ポリマーとエマルジョンとの相互の結合作用が弱く、その結果として、塗膜の長期的耐水性、耐アルカリ性といった耐久性の面に問題を残す結果となっている。 そのため、無機ポリマーと有機ポリマー・エマルジョンとの結び付きを強固にすべく、これら両ポリマーのブレンド物にアルコキシシラン類を添加する方法や、アルコキシシラン基を持った重合性モノマーを使用する方法も提案されている。 ところが、これら方法で得られる塗料用組成物は、それ自体の安定性が悪く、従って、長期的な保存貯蔵に耐えられないなど、未だ満足できる性質が得られていないのが実情である。



【０００９】また、従来よりTg値の比較的高いポリマー・エマルジョンに多量の造膜助剤を添加することにより、室温でも造膜させ得る塗料が提案されていた。 この場合、比較的沸点の低い造膜助剤を使用すれば、多量の溶剤添加が必要となり、相対的に少量の造膜助剤より形成された塗膜はクラックも入りやすい。 また、沸点の高い造膜助剤を用いれば、得られた塗膜は数日を経た後においても依然として「ベタツキ」が残り、耐汚染性の悪さが露呈するという具合である。 このように、造膜助剤の選択は難しく、造膜性と塗膜性能の両立は困難であった。

【００１０】

【課題を解決するための手段】本発明者は、上述した従来技術での課題に鑑み、耐久性、耐汚染性に優れた塗膜を形成する有用な水分散性被覆組成物を得るべく鋭意研究した結果、本発明を完成するに至ったものである。

【００１１】そして、本願発明の要旨とするところは、陰イオン界面活性剤および／または非イオン界面活性剤の存在下、水系媒質中にてモノマー混合物と乳化共重合せしめて得られる、50℃以上のTg値（ポリマーのガラス転移点）を有する水分散性被覆組成物であって、前記モノマー混合物が、樹脂量に対して(a) 70～99.9重量%のエチレン性不飽和モノマー、および前記(a) エチレン性不飽和モノマーと共重合可能な(b) 0.1～10重量%の水溶性エチレン性不飽和モノマーを含み、そして、前記組成物が、樹脂量に対して 0.5～10重量%であり、かつ混合重量比が２／８～８／２のエチレングリコール誘導体とジエチレングリコール誘導体の混合物を造膜助剤としてさらに含む、ことを特徴とした水分散性被覆組成物にある。

【００１２】

【発明の実施の形態】本発明の水分散性被覆組成物では、水を主成分とする分散媒にポリマー系分散質が分散したものを使用する。

【００１３】分散質の平均粒子径は、通常10～ 500nmの範囲とする。 これはすなわち、粒子径が10nm以下のものを得ようとすると、粘度などの関係上、低固形分になる傾向があり、塗料ベースとしては実用的でないこと、

また、粒子径が 500nmより大きくなると、耐久性の低下をきたすことを考慮して決定した。

【００１４】次に、乳化重合方法については特に限定された方法はなく、例えば、水、乳化剤、開始剤の存在下でラジカル重合可能な、(a) エチレン性不飽和モノマーおよび(b) 水溶性エチレン性不飽和モノマーの混合物を滴下するモノマー滴下法；ラジカル重合可能な、(a) エチレン性不飽和モノマーおよび(b) 水溶性エチレン性不飽和モノマーの混合物を、水、乳化剤の存在下で乳化し、それを滴下しながら重合を行うプレエマルジョン法；あるいは、水、乳化剤、開始剤、(a) エチレン性不飽和モノマーおよび(b) 水溶性エチレン性不飽和モノマーの混合物すべての存在下でラジカル重合を行う一浴重合法などが、本発明において使用できる。

【００１５】また、モノマー滴下法、プレエマルジョン法では、総滴下量の１～50重量%、好ましくは、３～30重量%を重合開始時に予め添加できる。　安全性の面からすれば、モノマー滴下法、プレエマルジョン法が好ましく、プレエマルジョン法の方が特に好ましい。

【００１６】本発明に適用可能な上記(a) エチレン性不飽和モノマーとしては、例えば、アクリル酸メチル、アクリル酸エチル、アクリル酸プロピル、アクリル酸ブチル、アクリル酸ヘキシル、アクリル酸シクロヘキシル、アクリル酸2-エチルヘキシル、アクリル酸ラウリル、アクリル酸ステアリル、メタクリル酸メチル、メタクリル酸エチル、メタクリル酸プロピル、メタクリル酸ブチル、メタクリル酸ヘキシル、メタクリル酸シクロヘキシル、メタクリル酸2-エチルヘキシル、メタクリル酸ラウリル、メタクリル酸ステアリルなどの（メタ）アクリル酸のＣ１～Ｃ24のアルキルまたはシクロアルキルエステル；ヒドロキシエチルアクリレート、ヒドロキシエチルメタクリレート、ヒドロキシプロピルアクリレート、ヒドロキシプロピルメタクリレートなどの（メタ）アクリル酸のＣ２～Ｃ８のヒドロキシアルキルエステル；スチレン、ビニルトルエン、α－メチルスチレン、Ｎ－ビニルピロリドン、ビニルピリジンなどの芳香族不飽和モノマー；グリシジルアクリレート、グリシジルメタクリレートなどのエポキシ基含有の（メタ）アクリル酸エステル；アクリル酸1-メチル2-イミダゾリドン、アクリル酸1-エチル2-イミダゾリドン、メタクリル酸1-メチル2-イミダゾリドン、メタクリル酸1-エチル2-イミダゾリドンなどの（メタ）アクリル酸のイミダゾール環含有のＣ１～Ｃ24のアルキルエステル；アクリル酸1-メチル2-ピロリドン、アクリル酸1-エチル2-ピロリドン、メタクリル酸1-メチル2-ピロリドン、メタクリル酸1-エチル2-ピロリドンなどの（メタ）アクリル酸のピロール環含有のＣ１～Ｃ24のアルキルエステル；アクリル酸1-メチル2-オキサゾリドン、アクリル酸1-エチル2-オキサゾリドン、メタクリル酸1-メチル2-オキサゾリドン、メタクリル酸1-エチル2-オキサゾリドンなどの（メタ）アクリル酸のオキサゾール環含有のＣ１～Ｃ24のアルキルエステル；または前述した化合物の一種以上の組み合わせなどがある。

【００１７】さらに、本発明に適用可能な上記(b) 水溶性エチレン性不飽和モノマーとしては、例えば、アクリル酸、メタクリル酸、マレイン酸、クロトン酸、ポリオキシエチレン鎖を有する（メタ）アクリレート；アクリルアミド、Ｎ－メチロールアクリルアミド、Ｎ－ブトキシメチルアクリルアミド；または前述した化合物の一種以上の組み合わせなどがある。

【００１８】次に、上記 (a)エチレン性不飽和モノマーおよび(b) 水溶性エチレン性不飽和モノマーの使用量としては、全樹脂量に対して下記の範囲の量を選択する。

すなわち、

(a) エチレン性不飽和モノマー：90～99.5重量%、好ましくは95～99重量%、および

(b) 水溶性エチレン性不飽和モノマー：0.5 ～10重量%、好ましくは１～５重量%、とする。

【００１９】本発明で使用される乳化剤としては、アニオン系界面活性剤、ノニオン系界面活性剤、両性界面活性剤などが挙げられる。　具体的には、アニオン系界面活性剤として、例えば、高級アルコールの脂肪酸エステル塩、アルキルベンゼンスルホン酸塩、脂肪族スルホン酸塩などが、また、ノニオン系界面活性剤として、ポリエチレングリコールのアルキルエステル型、アルキルエーテル型、アルキルフェニルエーテル型などが、さらに、両性界面活性剤として、アニオン部分をカルボン酸塩、スルホン酸塩、硫酸エステル塩、そしてカチオン部分をアミン塩、第４級アンモニウム塩とした界面活性剤が挙げられる。　また、上記界面活性剤に重合性官能基を導入した反応性乳化剤なども使用できる。　これら界面活性剤の使用割合としては、樹脂に対して 1.2～ 2.5重量%とすることが良好な耐久性を得る上で好ましい。

【００２０】本発明で使用されるラジカル重合開始剤としては、熱または還元性物質などによりラジカル分解してモノマーへの付加重合を開始せしめる、水溶性または油溶性の過硫酸塩、過酸化物、アゾ化合物などが使用できる。　例えば、過硫酸カリウム、過硫酸ナトリウム、過硫酸アンモニウム、過酸化水素、ｔ－ブチルハイドロパーオキサイド、ｔ－ブチルパーオキシベンゾエート、2,2-アゾビスイソブチロニトリル、2,2-アゾビス（2-ジアミノプロパン）ハイドロクロライドなどがあり、取扱いの容易性からして、水溶性のものが特に好ましく、樹脂に対して0.05～２重量%、好ましくは 0.1～ 0.5重量%の量が添加される。　なお、重合速度の促進、低温での重合を行う時は、重亜硫酸ナトリウム、塩化第一鉄、アスコルビン酸塩、ロンガリットなどの還元剤を、ラジカル重合開始剤と組み合わせて使用することもできる。

また、分子量の調節のため、ドデシルメルカプタンなどの連鎖移動剤を添加することも可能である。　本発明

で重合される水性エマルジョンは、水性エマルジョン中のカルボキシル基を中和しなくても安定な分散性を示すが、長期にわたって分散性を保つために、例えば、アンモニア、水酸化ナトリウム、水酸化カリウム、アミン類を用いてpHを6～10の範囲に調節できる。

【0021】本発明で使用される造膜助剤としては、ブチルセロソルブ、エチルセルソルブなどのエチレングリコールモノアルキルエーテルのようなエチレングリコール誘導体とエチルカルビトール、ブチルカルビトールなどのジエチレングリコールモノアルキルエーテル、ジエチレングリコールジエチルエーテルなどのジエチレングリコールジアルキルエーテルのようなジエチレングリコール誘導体との混合物が挙げられる。 これらの混合割合（重量比）は、エチレングリコール誘導体／ジエチレングリコール誘導体として、2／8～8／2の範囲で、好ましくは、3／7～7／3とする。 また、エチレングリコール誘導体、ジエチレングリコール誘導体は任意の比率で数種類併用することもできる。

【0022】これらの使用割合としては、良好な耐久性を得るとの観点から、樹脂に対して0.5～10重量%、好ましくは1～5重量%添加する。

【0023】また、本発明の水性エマルジョンは、通常水系塗料などに添加される成分、例えば、増粘剤、消泡剤、顔料、分散剤、染料、防腐剤なども添加できる。

【0024】

【実施例】次に、実施例と比較例を示して本発明を具体的に説明するが、本願発明がこれら開示によって限定的に解釈されるべきでないことは勿論である。 なお、以下の実施例と比較例での部および%の単位は、特に断りの無い限り、すべて重量表示による。

【0025】A．試料の調製

実施例1

撹拌機、還流冷却器、滴下戸斗、温度計を備えた2lの＊4つ口フラスコに水30部、過硫酸カリウム(KPS)0.2部を仕込み、撹拌下、系内を窒素ガスで置換し、85℃に昇温した。 同温度にて、水17部、界面活性剤4部に、メタクリル酸メチル38部、アクリル酸2-エチルヘキシル6部、アクリル酸 0.6部を混合することによって得たエマルジョンを、3時間かけて滴下し、さらに、85℃で2時間維持したあと、室温まで冷却した。 さらに、造膜助剤としてブチルセロソルブ 1.5部とジエチレングリコールジエチルエーテル 1.5部の混合液を添加し、25%アンモニア水溶液でpHを8に調整して目的とする水性エマルジョン（固形分約40%）を得た。

【0026】実施例2

造膜助剤としてブチルセロソルブ 1.5部とジエチレングリコールエチルメチルエーテル 1.5部の混合液を添加した以外は、実施例1と同様の操作で試料を調製した（固形分約40%）。

【0027】比較例1

モノマー組成を、メタクリル酸メチル30部、アクリル酸2-エチルヘキシル14部とした以外は、実施例1と同様の操作で試料を調製した（固形分約40%）。

【0028】比較例2

造膜助剤としてブチルセロソルブ3部を添加した以外は、実施例1と同様の操作で試料を調製した（固形分約40%）。

【0029】比較例3

造膜助剤としてジエチレングリコールジエチルエーテル3部を添加した以外は、実施例1と同様の操作で試料を調製した（固形分約40%）。

【0030】実施例1－2および比較例1－3で得られた組成物の詳細を、下記表1にまとめた。

【0031】

【表1】

| 使用原料 | 実施例 | | 比較例 | | |
|---|---|---|---|---|---|
| | 1 | 2 | 1 | 2 | 3 |
| 脱イオン水 | 47 | 47 | 47 | 47 | 47 |
| 界面活性剤*1 | 4 | 4 | 4 | 4 | 4 |
| メタクリル酸メチル | 38 | 38 | 30 | 38 | 38 |
| アクリル酸2-エチルヘキシル | 6 | 6 | 14 | 6 | 6 |
| アクリル酸 | 0.6 | 0.6 | 0.6 | 0.6 | 0.6 |
| 造膜助剤*2 | 3 | 3 | 3 | 3 | 3 |
| | BMG(1.5)<br>DBDG(1.5) | BMG(1.5)<br>EMDG(1.5) | BMG(1.5)<br>EMDG(1.5) | BMG(3.0)<br>-------- | --------<br>EMDG(3.0) |
| Tg値（計算値：℃） | 70 | 70 | 35 | 70 | 70 |

*1 界面活性剤：レベノールWZ（花王製）
*2 BMG:ブチルセロソルブ、DBDG: ジエチレングリコールジエチルエーテル、
EMDG: ジエチレングリコールエチルメチルエーテル

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【００３２】Ｂ．水分散性被覆組成物の諸特性の検定
次に、実施例１－２および比較例１－３で得られた水分散性被覆組成物を、スレート板に塗布量約100g/m² でスプレー塗装した後、 120℃で３分間乾燥させて、以下の試験に供する皮膜試料を得た。

【００３３】１．フィルム形成性
スレート板に形成された皮膜のクラックの有無を、光学顕微鏡（倍率： 200倍）にて観察した。

【００３４】２．皮膜の耐水性
スレート板に形成された皮膜を、60℃の温水に４時間浸漬させて、その後、水で急冷し、室温で乾燥させた後の塗膜の状態を、目視判定した。

【００３５】３．皮膜の耐汚染性（1）
スレート板に形成された皮膜上に、白色ワセリンにカーボンブラックを10%混入して調製した汚染物質の約１ｇ程度を、布に付けて均等の力で10往復擦り込み、その後、40℃で一週間養生させる。 養生後、汚染物質を清浄な布で拭き取り、水で十分に洗浄した後に、室温で乾燥させる。 乾燥後に皮膜の状態を、目視判定した。

【００３６】４．皮膜の耐汚染性（2）
スレート板に形成された皮膜上に赤インキ10%溶液をスポット状に置き、１時間放置する。 その後、ガーゼでインキを拭き取り、汚染性の程度を、目視判定した。

【００３７】以上の試験１．～４．の試験結果を、下記表２にまとめた。

【００３８】
【表２】

| 試験項目（試験 No.） | 実施例 1 | 実施例 2 | 比較例 1 | 比較例 2 | 比較例 3 |
|---|---|---|---|---|---|
| フィルム形成性（1） | ○ | ○ | ○ | × | ○ |
| 耐水性（2） | ◎ | ◎ | × | --- | △ |
| 耐汚染性(3) | ◎ | ◎ | △ | --- | △ |
| 耐汚染性(4) | ◎ | ◎ | △ | --- | × |

凡　例：フィルム形成性(1) …… ○：クラック無し
×：クラックが認められる

耐水性（2） …… ◎：透明性に変化無し
○：わずかに青白さが認められる
△：白化が認められる
×：全面白化、不透明化

耐汚染性(3) …… ◎：痕跡が全く認められない
○：わずかに痕跡が認められる
△：痕跡が認められる
×：痕跡がくっきり残っている

耐汚染性(4) …… ◎：インキ跡が全く認められない
○：わずかにインキ跡が認められる
△：インキ跡が認められる
×：インキ跡がくっきり残っている

【００３９】
【発明の効果】上記した一連の結果から明らかな通り、本発明により、所期の目的であった、優れた耐水性、耐汚染性を備えた皮膜を形成する水分散性被覆組成物の提供が実現されたのである。

【００４０】また、本発明の水分散性被覆組成物は、従来の水分散性被覆組成物の場合と同様の製造装置、製造条件によって、容易にかつ安価に工業規模で製造でき、有機溶剤型塗料の置換品としてコーティング剤市場に安定して大量に提供できるなどの効果をも奏するものである。

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for

# WEST

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## Summary

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| JP410007976A | 6 | 6 | 0 | 1 |
| Total (1) | 6 | 6 | 0 | - |

felts.

Detailed Description Text (3):
The present composition uses a combination of a cleaning solvent and a surfactant to prevent the deposition of contaminants from resinous pulp and/or recycled pulp in the felt press section of a pulp and paper process. The cleaning solvent comprises a blend of aromatic hydrocarbons containing between nine and eleven carbon atoms that preferably has a flash point of greater than 140.degree. F. Although the cleaning solvent includes aromatic hydrocarbons containing between nine and eleven carbon atoms, it can also include aromatic hydrocarbons having less than nine or carbon atoms or having more than eleven carbon atoms in accordance with the invention. Preferably, the cleaning solvent includes at least about 95% aromatic hydrocarbons, more preferably, more than about 98% aromatic hydrocarbons. The cleaning solvent (and the composition itself) is preferably substantially free of naphthalene (e.g. less than 1%). In addition, the present composition effectively cleans the pressing equipment while using a minimal amount of VOC's. An exemplary cleaning solvent for use in the invention is SURE SOL.RTM. 150ND, a C9-C11, naphthalene-depleted mononuclear aromatic solvent commercially available from Koch Specialty Chemical Company in Houston, Tex. (USA), having a specific gravity of 0.8796, a flash point of about 145.degree. F. and greater than 98% aromatic hydrocarbons.

Detailed Description Text (4):
The surfactant used in the present composition is an alcohol ethoxylate. The alcohol ethoxylates used in the invention typically include between 6 and 16 moles ethylene oxide (EO), preferably between 8 and 10 moles ethylene oxide, per mole of alcohol. Preferably, the alcohol ethoxylates are branched or secondary alcohol ethoxylates and more preferably, the alcohol ethoxylate is a tridecyl alcohol ethoxylate. An exemplary branched tridecyl alcohol ethoxylate having 8.5 moles ethylene oxide per mole of alcohol is Rhodasurf TDA 8.5 available from Rhodia in Cranbury, N.J. (USA). The surfactant used in the composition (and the composition itself) is substantially free of nonylphenol ethoxylates and other alkylphenol ethoxylates. In addition, the composition is preferably substantially free of other environmentally undesirable SARA reportable compounds.

Detailed Description Text (5):
The cleaning solvent and the surfactant are combined in the composition of the invention such that the mass ratio of cleaning solvent to alcohol ethoxylate surfactant in the present composition is preferably from about 1:10 to about 10:1, more preferably from about 1:3 to about 3:1. A particularly useful ratio of cleaning solvent to alcohol ethoxylate surfactant for removing pitch is 1:2.

Detailed Description Text (6):
In addition to the cleaning solvent and the alcohol ethoxylate, the present composition can also include other components. For example, the composition typically includes one or more formulation solvents to provide increased stability to the composition to prevent it from gelling or separating in storage. The formulation solvents are preferably substantially free of naphthalene and other undesirable compounds. Exemplary formulation solvents include polypropylene glycols, polyethylene glycols, d-limonene, propylene glycol n-butyl ethers, propylene glycol monomethyl ethers, dipropylene glycol monomethyl ethers, ethylene glycol monobutyl ethers, branched alcohol acetic acid esters (e.g. EXXATE.RTM. 800 from Exxon), aliphatic solvents and solvent blends, N-methyl-2-pyrrolidone, tetrahydrofurfuryl alcohols, and diisopropyl/triisopropylbiphenyl solvents. Water is also generally added to the formulation to provide increased flowability to the composition. Furthermore, the composition can also include additional non-alkylphenol surfactants to provide increased stability, e.g., in the presence of high concentrations of calcium ions. Buffers can also be included in the composition to provide a desired pH for use in the press felt section. The present composition can further include other additives known in the art for use in pulp and paper processes.

Detailed Description Text (7):
The present composition preferably includes from 5 to 80% of the aromatic hydrocarbon cleaning solvent, from 15 to 90% of the alcohol ethoxylate surfactant, from 0 to 15% of the formulation solvent, from 0 to 20% water, from 0 to 2% of a second surfactant, and from 0 to 5% of buffers and other additives, on a mass basis.

More preferably, the composition includes from 15 to 45% of the aromatic hydrocarbon cleaning solvent, from 40 to 70% of the alcohol ethoxylate surfactant, from 2 to 12% of the formulation solvent, from 2 to 15% water, from 0 to 1% of the second surfactant, and from 0 to 5% of buffers and other additives.

Detailed Description Text (15):
In accordance with the invention, the advancing felts 36, 38, 40 and 42 are continuously washed with a diluted form of the present composition after contacting the paper stock material 34. The present composition is diluted in water such that the composition as applied includes a deposition-inhibiting amount of the composition. Preferably, the diluted composition includes from 100 ppm to 10,000 ppm of the cleaning composition in water.

Detailed Description Text (16):
In FIG. 1, the diluted composition is applied to the felts 36, 38, 40 and 42 using any suitable apparatus such as chemical showers 110, 112, 114 and 116. Preferably, the chemical showers 110, 112, 114 and 116 are fan showers that apply the composition across the advancing felts 36, 38, 40 and 42. In addition, high-pressure showers can be used to mechanically dislodge the contaminants on the surface of the felts 36, 38, 40 and 42. Moreover, the diluted composition is preferably applied to the felts 36, 38, 40 and 42 using water at an elevated temperature of between about 120.degree. F. and 180.degree. F.

Detailed Description Text (17):
The diluted composition breaks down and loosens the contaminants deposited on the felts 36, 38, 40 and 42. Once the diluted composition has been applied to the felts 36, 38, 40 and 42, the felts are advanced to uhle boxes 118, 120, 122, 124, 126 and 128, that apply suction to the felts and remove both the water and the contaminants loosened by the present composition from the felts. Typically, after the diluted composition is applied to the felts 36, 38, 40 and 42, the felts are normally lubricated prior to advancing to the uhle boxes 118, 120, 122, 124, 126 and 128 using lube showers 130, 132, 134 and 136, thereby minimizing felt wear at the uhle boxes. In addition, the press section 10 can include caustic showers 138, 140 and 142 and needle showers 144, 146, 148 and 150 that further loosen contaminants on the surface of the felts 36, 38, 40 and 42. The forming section 12 can also include a needle shower 152 to remove contaminants from the inner wire mesh 24. Although not illustrated in FIG. 1, the present composition can also be applied to the rolls and uhle boxes to loosen or break down contaminants deposited on this equipment.

Detailed Description Text (22):
In FIG. 2, the diluted composition is applied to the felts 162, 164 and 166 using any suitable apparatus such as fan showers 230, 232 and 234, preferably at an elevated temperature. Once the diluted composition has been applied to the felts 162, 164 and 166, the felts are advanced to uhle boxes 236, 238, 240, 242, 244 and 246 that apply suction to the felts and remove both the water from the felts and the contaminants loosened by the present composition. After the diluted composition is applied to the felts 162, 164 and 166 and prior to advancing to the uhle boxes 236, 238, 240, 242, 244 and 246, the felts are normally lubricated using lube showers 248, 250, 252, 254, 256 and 258. Although not illustrated in FIG. 2, the press section 160 can also include additional showers that further loosen contaminants on the surface of the felts 162, 164 and 166, and the present composition can also be applied to the rolls and uhle boxes to loosen or break down contaminants deposited on this equipment.

Detailed Description Text (26):
Actual fresh pitch from deposits in a thermomechanical pulp (TMP) mill were smeared onto MYLAR.RTM. coupons. MYLAR.RTM. was chosen because its surface energy and interactions are known to be similar to that of nylon, which is a common component of paper making felts. MYLAR.RTM. is also related in behavior to other surfaces like polyethylene and rubber used for uhle box covers and rolls. The coupons were mounted in the shear zone of a blender and 200 ml of water at a temperature of 120.degree. F. and the chemicals listed in Table 1 were added to the blender. The blender was run at a low setting for 5 minutes. The performance of the formulation was determined by measuring the fraction of the applied pitch that was removed. This was determined by weighing the coupons before applying the mill pitch, after applying

the pitch, and after exposure to the cleaning chemicals. Table 1 below shows the results of these tests.

Detailed Description Text (27):
As shown by the above data, the preferred surfactant/solvent combinations comprising the C9-C11 aromatic solvent and either the branched tridecyl alcohol ethoxylate or the secondary alcohol ethoxylate produced synergistic results when removing pitch, i.e., the actual combination of the surfactant and the solvent performed better than the expected additive effect for these components. In addition, some of the primary alcohol ethoxylates produced synergistic results when combined with the C9-C11 aromatic solvent. Moreover, the preferred branched and secondary alcohol ethoxylate surfactants were also more effective alone and in combination with the preferred solvent of the invention than the nonylphenol ethoxylates. The above tests show that the combination of the surfactants and solvents of the present invention are effective, even at very low concentrations.

Detailed Description Text (32):
Uniform areas of MYLAR.RTM. coupons were coated with a retail automotive grease that was gray to black in color. The grease was used to simulate newsprint ink, which is basically a hydrocarbon oil with carbon black dispersed in it. These coated MYLAR.RTM. coupons were suspended in beakers of test cleaning solutions (50,000 ppm) that were in a temperature controlled water bath at 55.degree. C. The solutions were further provided with moderate agitation using magnetic stirrers. At the end of a 15-minute exposure period, the MYLAR.RTM. coupons were removed and gently rinsed with cold water. The degree of grease removal was determined by placing the coupons on a white paper backing and measuring the brightness of the area that was previously coated with grease. The higher the measured brightness the more completely the grease had been removed. The results of this test are provided in Table 3 below.

Detailed Description Text (33):
As shown by the above data, the tridecyl alcohol ethoxylate alone or in combination with the preferred solvent was superior to the prior art nonylphenol ethoxylates as well as many other surfactants of common use in removing ink-type deposits. In addition, the aromatic solvents with flash points of 140.degree. F. or greater were more effective than many other solvents in common use at removing ink-type deposits.

Detailed Description Text (39):
9% Water

Detailed Description Text (40):
This composition was used in the press felt section of a paper machine that made paper from a combination of resinous and recycled pulp sources and that previously operated using a nonylphenol-containing composition. The composition of the invention was found to be superior to the nonylphenol-containing composition at removing pitch and other deposits from the press felt section. Although the composition of the invention displayed good stability, it experienced some flocking and gelling when it was diluted with hot water containing high concentrations of calcium ions.

Detailed Description Text (47):
8.4% Water

Detailed Description Paragraph Table (1):
TABLE 1 Performance of Surfactant and Surfactant/Solvent Combinations in Removing Pitch % Removal of Pitch 3,000 ppm 3,000 ppm 2,000 ppm 1,000 ppm Surf- Surfactant Surfactant Surfactant actant 3,000 ppm 2,000 ppm 1,000 ppm Chemical Name only Solvent* Solvent* Solvent* Nonylphenol Ethoxylate with 20% 92% 88% 80% 9 mole EO (comparative) Branched Tridecyl Alcohol 54% 99% 96% 90% Ethoxylate with 8.5 moles EO 12-14 Carbon Secondary 57% 94% 88% Alcohol Ethoxylate with 9 moles EO 12-15 Carbon linear alcohol 16% 48% ethoxylate with 3 moles EO 12-14 Carbon linear alcohol 31% 74% 50% ethoxylate with 7 moles EO 12-16 Carbon linear alcohol 55% 88% 72% ethoxylate with 7 moles EO 14 Carbon linear alcohol 67% 50% 38% ethoxylate with 7 moles EO 12-14 Carbon linear alcohol 0% 90% 91% 72% ethoxylate with 9 moles EO 12-16

Carbon linear alcohol 0% 96% 89% ethoxylate with 9 moles EO 12-16 Carbon linear alcohol 0% 92% 86% ethoxylate with 11 moles EO 12-16 Carbon linear alcohol 5% 81% 84% ethoxylate with 15 moles EO Alkyl diphenyl oxide 6% 60% disulfonate DOWFAX .RTM. 2A1 from Dow (comparative) Phosphate ester 8% 97% RHODAFAC .RTM. RE-610 from Rhodia (comparative) Phosphate ester 18% 74% MAYPHOS .RTM. 8135 from BASF (comparative) *The solvent used was SURE SOL .RTM. 150ND, a C 9-11 naphthalene-depleted, mononuclear aromatic solvent commercially available from Koch Specialty Chemical Company. 2500 ppm of the SURE SOL .RTM. 150ND solvent was tested alone (without surfactants) and resulted in 42% removal. In addition, 2500 ppm tetra hydro furfural alcohol (THFA) was tested alone (without surfactants) and resulted in 6% removal, significantly less than the SURE SOL .RTM. 150ND solvent.

Detailed Description Paragraph Table (3):
TABLE 3 Performance of Components and Blends in Removing Ink Type Contaminants (Grease) Treatment Measured Brightness Nonylphenol Ethoxylate (9 moles) 18.7 Nonylphenol Ethoxylate/SURE SOL .RTM. 20.5 150ND 2:1 Blend Branched Tridecyl Alcohol Ethoxylate (8.5 46.4 moles) Branched Tridecyl Alcohol Ethoxylate/ 65.2 SURE SOL .RTM. 150ND 2:1 Blend Solvent Only SURE SOL .RTM. 205 68.4 SURE SOL .RTM. 150ND 67.8 EXXATE .RTM. 800 66.7 d-Limonene 66.0 Aliphatic 140 Solvent 67.3 DOWANOL .RTM. PnB 59.4 Propylene Glycol 16.4 DOWANOL .RTM. DPM 15.9 DOWANOL .RTM. PM 15.9 m-Pyrol 15.6 Tetrahydrofurfuryl alcohol 13.8 Butyl CELLOSOLVE .RTM. 13.4 Surfactant Only Alkyl phenyl oxide disulfonate 13.7 DOWFAX .RTM. 2A1 from Dow 14-16 Olefin Sulfonate BIO TERGE .RTM. 13.7 AS-40 from Stepan Alkyl Benzene Sulfonate BIOSOFT .RTM. 12.7 D-40 from Stepan Phosphate ester with undisclosed structure 11.1 from BASF MAYPHOS .RTM. 8135 SURE SOL .RTM. 205: Mixed aromatic solvent with flash point of 205 F. from Koch EXXATE .RTM. 800: Branched alcohol acetic acid ester with Flash point of 171 F. from Exxon Aliphatic 140 Solvent: Generic aliphatic solvent blend with flash point of 140 F. from Ashland DOWANOL .RTM. PnB: Propylene glycol n-butyl ether from Dow DOWANOL .RTM. DPM: Dipropylene glycol monomethyl ether from Dow DOWANOL .RTM. PM: Propylene glycol monomethyl ether from Dow m-Pyrol: N-methyl-2-pyrrolidone from International Specialty Products Butyl CELLOSOLVE .RTM.: Ethylene glycol monobutyl ether from Union Carbide

CLAIMS:

6. The composition according to claim 1, further comprising water.

11. The composition according to claim 1, further comprising an additional surfactant.

18. The composition according to claim 12, further comprising water.

23. The composition according to claim 12, further comprising an additional surfactant.

24. A composition for inhibiting the deposition of pitch and other deposits on pulp and paper machinery comprising:

a cleaning solvent comprising a blend of aromatic hydrocarbons containing between nine and eleven carbon atoms, having a flash point of greater than 140.degree. F., and being substantially free of naphthalene;

from 0 to 20% water; and

a surfactant selected from the group consisting of branched alcohol ethoxylates, secondary alcohol ethoxylates, and mixtures thereof;

said composition being substantially free of alkyl phenols and alkylphenol ethoxylates.

25. The composition according to claim 24, comprising from 5 to 80% of the cleaning solvent, from 15 to 90% of the surfactant, from 0 to 15% of a formulation solvent, from 0 to 2% of a second surfactant, and from 0 to 5% of a buffer.

27. The composition according to claim 26, further comprising water.

28. The composition according to claim 27, comprising from 15 to 45% of the cleaning solvent, from 40 to 70% of the surfactant, from 2 to 12% of the formulation solvent, from 2 to 15% water, from 0 to 1% of a second surfactant, and from 0 to 5% of a buffer.

L12: Entry 32 of 90 File: USPT Jan 16, 2001

DOCUMENT-IDENTIFIER: US 6173776 B1
TITLE: Cleaning compositions for oil and gas wells, lines, casings, formations and equipment and methods of use

Brief Summary Text (5):
Typical prior art methods consist of four major types of products. The first method uses aggressive blends of aromatic and/or aliphatic petroleum hydrocarbons or halogenated hydrocarbons. These products may or may not contain surfactants to allow the products to mix with and emulsify in water to increase cleaning efficiency. The presence of water decreases the efficiency of these compositions significantly. Pure solvent blends are immiscible in water and cannot penetrate to the soils. The addition of emulsifiers allows the solvents to blend with the water, but the resulting solvent/emulsion system is not as efficient a cleaner as the pure solvent.

Brief Summary Text (7):
The third method is predicated on water-based alkaline, hard-surface cleaners. These cleaners generally incorporate alkaline builders, water-soluble solvents, such as glycol ethers, alcohols, and surfactants. Alkaline builders consist of hydroxide, carbonate, phosphate, and silicate. Water-soluble solvents typically consist of ethylene glycol, diethylene glycol, propylene glycol and dipropylene glycol ethers. Typical surfactants are the categories of alkyl phenol ethoxylates, linear alcohol ethoxylates, or alkyl sulfonates, amphoterics, and fatty acid soaps of alkanolamides. The cleaning efficiency of these alkaline compositions on paraffins and other oil-based soils is typically much lower than that of solvent blends. Further, these compositions are not effective for removing scale.

Brief Summary Text (9):
Typical prior art compositions employ solvents of aromatic and/or aliphatic petroleum hydrocarbons or halogenated hydrocarbons. These solvents produce vapor emissions, commonly known as "VOCs" (volatile organic compounds), which typically are toxic. Other cleaning compositions require little or no VOCs, but require high levels of caustic and/or phosphates which cause problems in transportation, use, and disposal.

Brief Summary Text (11):
The bacteria-based cleaning solution also has other disadvantages. The treatment requires that the well be shut down for two weeks to allow the establishment of a bacterial colony. When pumping is resumed, the bacteria are removed with the oil, which results in rapid depletion of the colony and retreatment is required.

Brief Summary Text (13):
An object of the present invention is to provide a method of cleaning gas and oil wells, equipment, well casing perforations, and surrounding formations, pumps, as well as lines, pipes, and the like using an effective cleaning composition which is free of objectionable petroleum-derived or halogenated hydrocarbon and alkaline solvents and useful in removing most of the soils and scale encountered in gas- and oil-well cleaning operations.

Brief Summary Text (16):
A further object is to use a safe liquid cleaning composition containing fatty acid methyl esters and lower alkyl glycol ethers, which is effective in removing

paraffins, tars, related soils, and scale from gas- and oil-wells, casings and equipment.

Brief Summary Text (22):
A further object is a method to separate excess water from recovered crude petroleum oil.

Brief Summary Text (24):
The present invention is also directed to a method for separating excess water, sludge, or both, from recovered crude petroleum oil comprising adding to the crude petroleum oil a composition comprising about 40 to 99 wt % of a fatty acid alkyl ester blend and about 1 to 25 wt % of at least one lower alkyl glycol ether, and then agitating the mixture.

Detailed Description Text (3):
It was discovered that combining fatty acid alkyl ester blends with lower alkyl glycol ethers provides cleaning compositions having excellent properties, i.e., high flash point, good soil and paraffin penetration, and excellent protection against resoiling and scale buildup. The compositions used in the methods of the present invention are blended to provide low VOCs (less than 50%) and to provide a composition with a flash point above 200.degree. F. TCC (tag closed cup). The compositions are also safe and biodegradable.

Detailed Description Text (4):
The present invention is directed to methods for removing and preventing the buildup of paraffins, other related soils, and scale from gas and oil wells, hydrocarbon bearing formations, and recovery, pumping, storage, and transmission equipment by injecting into the wells and such equipment a cleaning composition comprising about 40 to 99% by weight of a fatty acid alkyl ester blend; and about 1 to 25% by weight of at least one lower alkyl glycol ether, with the remainder being suitable additives. Preferably the composition contains about 60 to 95% by weight, more preferably about 80 to 90% by weight, of the fatty acid alkyl ester blend, and preferably about 1 to 15% by weight of the lower alkyl glycol ether.

Detailed Description Text (12):
In other embodiments water is substituted for the oil. As with the oil, the water and composition may be mixed prior to introduction to the well or introduced separately. The water temperature may be ambient temperature or elevated to about 150-160.degree. F. Again, the mixture may be allowed to set, circulated, or both.

Detailed Description Text (14):
The composition may be introduced by means of injection pumps into off-shore gas or oil wells to reduce soils, particularly paraffin, or scale adhesion in well casings and transmission lines. In addition to the problems associated with land oil wells, off shore wells have the further problem of the ocean or sea water behaving as a coolant of the lines and contents between the bottom of the ocean and the platform. Thus off shore wells have a particular problem with paraffin buildup. To treat the lines, 40-50 gallons of the composition, for example, are dropped into the lines. Preferably, the composition is heated prior to being introduced into the lines.

Detailed Description Text (19):
1 to 25 wt. % of a lower alkyl glycol ether may be added as a penetrant, to reduce viscosity of the mixture, as a coupling agent, and/or to increase efficacy on hydrophilic soils. Examples of lower alkyl glycol ethers useful in the present composition include dipropylene glycol monomethyl ether, tripropylene glycol monomethyl ether, ethylene and diethylene glycol ether, methyl, ethyl, propyl and butyl ethers, such as ethylene glycol monobutyl ether, or mixtures thereof.

Detailed Description Text (20):
A polyoxyalkylene glycol ether may be present in the composition in amounts of about 1 to 40 wt. %, preferably 3 to 25 wt. %, and most preferably 3 to 10 wt. %. The actual amount utilized depends on the types of soil present in the equipment to be cleaned and on the water content of the well or equipment being cleaned. Preferably used are polyethylene glycol ethers and polypropylene glycol ethers having the formulas:

Detailed Description Text (22):
Preferably, at least 1 wt %, more preferably, 1.5 to 3 wt %, of antioxidants are included in the composition. Antioxidants suitable for the present invention include, but are not limited to, (BHT) 2,6di-tert-butyl-para-cresol, (BHA) 2,6-di-tert-butyl-para-anisole, Eastman inhibitor O A B M-oxalyl bis(benzylidenehydrazide), and Eastman DTBMA 2,5-di-tert-butylhydroquinone. A surfactant may also be added to the composition. Any surfactant suitable for use in cleaning oily soils may be used, such as ethoxylated nonylphenols, linear alcohol ethoxylates, alkanolamine salts of dodecylbenzene sulfonic acid, sulfosuccinates, phosphate esters, alcohol sulfates, quaternary ammonium compounds, amphoteric surfactants, alpha-olefm sulfonates, sorbitan, and fatty acid derivatives. The surfactant is added in an amount effective to perform as a wetting agent and emulsifier, and usually up to 10 wt. %, preferably 1-3 wt %, of the composition.

Detailed Description Text (23):
Commercial surfactants include the EXXATE series of surfactants obtained from EXXON. EXXATE 1000 is an acetic acid ester of C.sub.9 -C.sub.11 branched oxo-alcohol. DBE (DuPont) is a blend of 45-75 wt. % dimethyl glutarate, 10-25 wt. % dimethyl adipate, and 15-30 wt. % dimethyl succinate.

Detailed Description Text (26):
Amines such as morpholene, 1,3-dimethyl-2-imidazolidinone, 1,3 propanediamine, 2-amino-1,3- propanediol, and 3-amino propanol, and alkanolamines such as triethanolamine, diethanolamine, 2-aminomethyl propanol, and monoethanolamine act as dispersants for soils and solubilize fatty acids and oils. Amino acids, such as choline and choline hydroxide, provide nontoxic alternatives to monoethanolamine, and act as metal chelators, preferably, methyl or isobutylesters of C.sub.4 -C.sub.6 aliphatic dibasic esters and n-methyl-2 pyrrolidone. Preferably up to 5 wt % of n-methyl-2 pyrrolidone is included.

Detailed Description Text (27):
Other additives typically used in cleaning compositions may be used, including water softening agents, sequesterants, and corrosion inhibitors, which are added in amounts effective to perform their intended function. These additives and amounts thereof are well within the skill of the art. Suitable water softening agents include linear phosphates, styrene-maleic acid co-polymers, and polyacrylates. Suitable sequesterants include 1,3-dimethyl-2-imidazolidinone, 1-phenyl-3-isoheptyl-1,3-propanedione, and 2-hydroxy-5-nonylacetophenoneoxime. Examples of corrosion inhibitors include 2-aminomethyl propanol, diethylethanolamine benzotriazole, and methyl benzotriazole.

Detailed Description Text (29):
Typical treatment of a well requires pumping the cleaning solution into the well casing, and circulating the solution from 24 to 72 hours. A typical well has a 41/2" casing which extends down through the depth of the well. The 41/2" casing at the surface is capped by a well head. The well head has a fitting on top through which 23/8" tubing is inserted. The tubing can extend to the bottom of the well and can be removed in sections for cleaning and maintenance. There are two 2-inch ports on the side of the well head which support the back lines. The normal procedure consists of injecting 5 to 20 gallons of the cleaning solution into the casing side of the well (back lines), and circulating it up the central 23/8" tubing. The depth of the well, casing diameter, and anticipated amount and nature of the soils, determine the exact dosage needed. The flow of the cleaning solution is maintained for 24 to 72 hours to remove all soils. Depending on buildup, repeated treatments may be provided every 2 to 3 weeks.

Detailed Description Text (30):
Another embodiment of the present invention is directed to a method for separating excess water and sludge from recovered crude petroleum oil. Crude oil contaminated with sludge and/or water is not pure enough for resale. The water or sludge is generally removed by "rolling" the tank via injecting natural gas under pressure or by adding dry ice to cause agitation of the liquid. The process of oil/water or oil/sludge separation is slow unless an accelerator is added. Thus a further embodiment of the present invention comprises adding to the crude petroleum oil in

an amount to accelerate oil/water or oil/sludge separation, a composition comprising about 40 to 99 wt. % of a fatty acid alkyl ester blend and about 1 to 25 wt. % of at least one lower alkyl glycol ether and then agitating to mix.

Detailed Description Text (31):
Typically 1-100 gallons of composition are used to separate water and sludge from 100-400 barrels of oil. The composition is added to the tank and then the tank is "rolled" by introducing natural gas or carbon dioxide for about 24 hours. The use of the composition in the tank resulted in complete separation of the water, which was removed from the bottom of the holding tank. Standard indicator test strips or indicator reagents for water in oil indicated no detectable level of water.

Detailed Description Text (39):
A 700 foot well, experiencing problems with paraffin and sludge buildup, was treated with the composition. The composition was introduced in one of two methods. In the first method, five gallons of the mixture were poured down the 2" back lines and up the 23/8" central tubing. The second method was by chemical injection pump from a small holding tank or chemical pot. The composition was circulated for 48 hours. At the end of this time, operation of the well was resumed. Before cleaning, the daily production of oil was 1/4 of a barrel. After treatment, daily production increased to two barrels. After 60 days of operation, the tubing was pulled from the well and examined. It was found that the tubing was coated with the cleaning composition and that there was very little adhesion of paraffin and tars to the tubing. Production of oil was still nearly two barrels a day.

Detailed Description Text (49):
A composition of the type described in Example 3 was introduced into a 3,000 foot open well with a casing but containing no pump, rods or tubes in a process known as "swabbing." In this case, the well had been completely shut down due to paraffin and other hydrocarbon blockages. Five gallons of the composition was first poured into the well, followed by 5 barrels of water at ambient temperatures. The mixture was allowed to set for 48 hours. Thereafter, the fluid pressures in the well increased significantly and production of oil increased from 0 to 8 barrels per day.

Detailed Description Paragraph Table (2):
Fatty acid methyl ester 67.5% n-Butoxy polyalkylene glycol ether 20.0% Ethylene glycol monobutyl ether 4.5% Diethylene glycol monomethyl ether 1.0% Dipropylene glycol monomethyl ether 1.0% Tripropylene glycol monomethyl ether 1.0% Exxate 900 (Exxon) 1.0% Exxate 1000 (Exxon) 1.0% 1-Methyl 2-pyrrolidinone 1.0% Dibasic esters 1.0%

Detailed Description Paragraph Table (3):
Fatty acid methyl ester 86.0% Nonylphenol (9.5 mole Ethylene Oxide) 1.5% Nonylphenol (6.0 mole Ethylene Oxide) 1.5% Ethylene glycol monobutyl ether 1.0% Diethylene glycol monomethyl ether 1.0% Dipropylene glycol monomethyl ether 1.0% Tripropylene glycol monomethyl ether 1.0% Exxate 900 (Exxon) 1.0% Exxate 1000 (Exxon) 1.0% Exxate 3000 (Exxon) 1.0% 1-Methyl 2-pyrrolidinone 1.0% Dibasic esters 1.0%

CLAIMS:

5. A method according to claim 1 wherein the lower alkyl glycol ether is selected from the group consisting of ethylene glycol monobutyl ether, diethylene glycol monobutyl ether, dipropylene glycol monomethyl ether, tripropylene glycol monomethyl ether, and mixtures thereof.

11. A method according to claim 1 further comprising up to 10 wt % of a surfactant.

12. A method according to claim 11 wherein the surfactant is selected from the group of ethoxylated nonylphenols, linear alcohol ethoxylates, and alkanolamine salts of dodecylbenzene sulfonic acid.

22. A method according to claim 15 wherein the composition is mixed with water prior to introducing into the well.

23. A method according to claim 22 wherein about 10 to 200 gallons of the

composition is mixed with 40 barrels of water.

24. A method according to claim 22 wherein the water composition mixture is heated to about 150-160.degree. F. prior to introducing into the well.

25. A method according to claim 15 wherein the composition is first introduced into the well and then water is introduced into the well.

WEST

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L12: Entry 35 of 90 File: USPT Oct 10, 2000

DOCUMENT-IDENTIFIER: US 6130195 A
TITLE: Cleaning compositions and methods for cleaning using cyclic ethers and alkoxy methyl butanols

Brief Summary Text (8):
Other materials that can be added are one or more of the following materials: water, alcohols, esters, ethers, cyclic ethers, ketones, alkanes, terpenes, dibasic esters, glycol ethers, pyrollidones, or low or non ozone depleting chlorinated and chlorinated/fluorinated hydrocarbons.

Brief Summary Text (16):
The art in the mid 1990's progressed as aqueous and semi-aqueous materials became the major choice of replacement for ODP's. Many of the materials developed and selected were materials that usually had lower toxicity, volatility and higher flash points than common solvents. The art generally developed along three basic type of cleaning materials. These materials were water insoluble organics, water soluble inorganics and water soluble organics.

Brief Summary Text (17):
The development of water insoluble cleaning agents as ODP replacements took many new art forms, disclosed in many countries. Typically this art included the predominant use of aliphatic and aromatic hydrocarbons, terpene hydrocarbons, and water insoluble esters. These products usually were good agents to clean and solvate organic contaminants, however they had drawbacks in that they were difficult to rinse with water and had little effect on ionic or inorganic residues. In addition, being water insoluble they were limited in their application and could not be diluted with water for spray applications.

Brief Summary Text (18):
The art of water soluble inorganic materials has been well known for years, usually in low technology applications where gross contaminant removal was desired. The art was upgraded in the last 10 years as work was done to create new mixtures that had solvating and cleaning efficacy in high technology applications where ODP materials were used. The bulk of the inorganic materials used were alkali metal salts (usually sodium or potassium) which included hydroxides, carbonates, silicates, phosphates, and bicarbonates. Many of the inorganic mixtures also included the use of surfactants and water soluble organic solvents to assist in the cleaning application. Cleaning agents of this art usually were inexpensive and found application in many non critical cleaning applications. The drawback of this art is that the mixture usually had solubility for a narrow range of contaminants, and in most cases was ineffective against tough contaminants. Other issues concerned the high pH required of the mixture to effectively clean, concern of possible alkaline residues left on the substrate due to inadequate rinsing, and short bath life due to consumption of the agent by the contaminant.

Brief Summary Text (19):
The art of water soluble organic materials as ODP replacements was the third and most flexible route chosen as replacement materials. Typically the art included materials such as alcohols, ethers, esters, glycol ethers and pyrollidones. Most of the formulations that have been disclosed utilized these materials either alone or in combination with other solvents, alkalinity agents and or water. Most of the alcohols, esters and ethers selected that were water soluble typically had low

molecular weights that created flash point or volatility issues in the mixture. Glycol ethers were another choice, however toxicity concerns became an issue with ethylene based glycol ethers. The art in the 1990's tended to move to propylene based glycol ethers because of their lesser toxicity concern. These materials however were not as robust as cleaners as alcohols or ethylene based glycol ethers, and required selective formulation and/or higher concentrations of the materials. Pyrollidones were also used in the art, however their broad use was limited because of cost, toxicity concerns and the highly aggressive nature of the material to some substrate materials.

Brief Summary Text (20):
A major drawback of the water soluble materials was the constant balance that was required to make the formulation clean a broad range of contaminants. Typically materials and mixtures could be found that were effective on ionic or polar soils, but were not effective on non-polar soils or oils. In addition some water soluble materials were very aggressive to some substrate materials such as coatings and metals. Hence proper selection of water soluble base materials is a key parameter in obtaining effective cleaning mixtures that clean efficiently and exhibit superior results over a broad range of contaminants.

Brief Summary Text (23):
According to this invention, novel cleaning compositions are provided which contain a mixture of materials that have been found to be synergistic in cleaning a broad range of soils and contaminants. The mixture contains one or more compounds from the family described as a four-carbon cyclic ether, known in the art as a tetrahydrofuran ring. Four carbon cyclic ether solvents of the invention correspond to the following formula: ##STR3## Where R.sub.1 and R.sub.2 can be independently hydrogen, or 1 to 8 carbon length alkyl, alkoxy or ether groups. Preferred compounds of formula I are water soluble and exhibit flash points greater than 100.degree. F. (ca. 38.degree. C.).

Brief Summary Text (24):
The second required compound of the mixture contains one or more compounds from the family described as a 3-alkoxy-3-methyl butanol and corresponds to the following formula: R.sub.3 ##STR4## Where the OH group of the butanol can be attached to carbon position 1, 2 or 4, and R.sub.3 is hydrogen or 1 to 8 carbon length alkyl. Preferred compounds of formula II are water soluble and exhibit flash points greater than 100.degree. F.

Brief Summary Text (25):
Other optional compounds are materials that can be added to a mixture of the compounds of Formula I and Formula II that will maintain the pH of the mixture at greater than 6. The optional alkaline material is any material known to those skilled in the art that would cause the pH of the solution to be greater than 6. Materials such as alkaline hydroxides, carbonates, bicarbonates, and silicates, preferably those of the alkali or alkaline earth metals or ammonium; and nitrogen containing materials such as amines, alkanolamines, quaternary ammonium hydroxides and amides can be used in the present invention. The preferred compounds of the cleaning compositions are nitrogen containing compounds that also contain one hydroxyl group.

Brief Summary Text (26):
Other optional materials that can be added are one or more of the following materials: water, alcohols, esters, ethers, cyclic ethers, ketones, alkanes, terpenes, dibasic esters, glycol ethers, pyrollidones, or low or non ozone depleting chlorinated and chlorinated/fluorinated hydrocarbons. Preferred compounds that can be added are water soluble and exhibit flash points greater than 100.degree. F.

Brief Summary Text (27):
The compositions may also be enhanced by one skilled in the art by adding buffering agents, surfactants, chelating agents, colorants, dyes, fragrances, indicators, inhibitors, and other conventional ingredients.

Brief Summary Text (28):
More specifically, the cleaning composition of the invention generally has a pH

greater than 6.0, and contains effective amounts of the compounds of Formula I and Formula II.

Brief Summary Text (35):
Preferred compositions and methods for cleaning mixtures in accordance with this invention optionally contain effective amounts of materials that can be added to a mixture of the two above disclosed materials that will maintain the pH of the mixture at greater than 6. The optional alkaline material is any material known to those skilled in the art that would cause the pH of the solution to be greater than 6. Materials such as alkaline hydroxides, carbonates, bicarbonates, and silicates; and nitrogen containing materials such as amines, alkanolamines, quaternary ammonium hydroxides and amides can be used in the present invention. The preferred compounds of the cleaning compositions are nitrogen containing compounds that also contain one hydroxyl group. Most preferred are monoethanolamine, diethanolamine, triethanolamine, 1-amino-2-propanol, ethylenediamine, hexamethyldiamine, 1,3-pentanediamine, n-isopropyl hydroxylamine, and 2-methyl-pentamethylenediamine.

Brief Summary Text (36):
The materials of Formulas I and II useful as cleaning mixtures in accordance with this invention are soluble in various solvents, such as water, alcohols, aqueous inorganic hydroxides, esters, ethers, cyclic ethers, ketones, alkanes, terpenes, dibasic esters, glycol ethers, pyrrolidones, or low or non-ozone depleting chlorinated and chlorinated/fluorinated hydrocarbons. Thus, the composition or mixture utilized in the process of the invention, and which comprises one or more of the above-described compounds, may be dissolved in any one or more of the before-mentioned solvents as an additional component of the cleaning composition. The detailed description below provides a non-limiting disclosure of the additional components that may be selected. The compositions of the invention, thus, may also include one or more of the above-mentioned solvents. Aqueous and non aqueous solutions of tetrahydrofurfuryl alcohol, 3-methyl 3-methoxy-1-butanol and amines, alkaline agents containing 1 or more hydroxyl groups are preferred in the practice of the invention, but other solvents may be used in conjunction with those. The form the compositions are in when used for cleaning may vary from liquid at various temperatures, to vapor, to aerosol, or other dispersions appropriate for the components of the composition selected. Buffers, corrosion inhibitors and other additives may also be included in the cleaning compositions of the invention.

Brief Summary Text (39):
Contacting an article with a cleaning composition according to the invention may be through a conventional process or means known in the art that includes but is not limited to: wiping; spraying; immersing; high pressure spray agitation; ultrasonic agitation; vapor degreasing; and soaking. The equipment to perform these processes is known in the art or can be devised from other fields where applying a composition to a solid surface is involved. The process may be conducted at ambient temperature or up to the boiling point of the selected cleaning composition. Generally, temperature ranges from about 32.degree. F. (0.degree. C.) to about 230.degree. F. (110.degree. C.) are used. The temperature used may also be determined by the selection of the manner of contacting the cleaning composition to the surface to be cleaned. The process is most commonly conducted at atmospheric pressure, but may be conducted at elevated pressure, in a vacuum, or at lower than atmospheric pressure conditions.

Brief Summary Text (40):
The part or article is contacted with the desired cleaning composition for a sufficient period of time to essentially remove the contaminant or remove the desired amount of the contaminant. The part or article can also be called a "surface" that is to be cleaned. Depending on the nature of the article and the use to which it will be put, it may not be necessary for every detectable trace of a contaminant to be removed from the surface. The contaminant may be any unwanted or undesired materials in contact with the substrate surface and may include is not limited to oils, grease, dirt, flux, inks, coatings, photoresists, resins and polymers, present in an amount ranging from a residue to a clearly visible amount.

Brief Summary Text (41):
It may, in most instances, be necessary or desirable to rinse the cleaning

composition from the part or article with water or with one of the solvents listed above, or with any combination of water and solvents. One skilled in the art can devise numerous combinations of cleaning compositions and rinsing solutions from this disclosure and the known properties of the chemicals used. In addition, one skilled in the art can devise simple tests to determine the appropriate rinsing conditions for a cleaning composition selected. It is common in the art to select a rinsing solution that will effectively remove all of the cleaning agent or composition and allow the rinsing solution to dry from the part either through the use of moving air, heated air and/or natural evaporation. Compounds that affect the odor of a surface being cleaned, that inhibit the corrosion of the surface, or that act as a surfactant can also be added to the cleaning compositions or rinsing solutions and used in the cleaning methods.

Brief Summary Text (44):
Other materials that can be added to the composition and/or used in the method of the invention are one or more of the following materials: water; alcohols; esters; ethers; cyclic ethers; ketones; alkanes; terpenes; dibasic esters; glycol ethers; pyrrolidones; or low or non-ozone depleting chlorinated and chlorinated/fluorinated hydrocarbons. The resulting mixture may also be enhanced by one skilled at the art by the addition of buffering agents, surfactants, chelating agents, colorants, dyes, fragrances, indicators, inhibitors, and other conventional ingredients.

Brief Summary Text (45):
Preferably, an effective amount of water is added to the solution to increase cleaning efficiency, decrease flash point, modify viscosity, or modify the solution's aggressiveness to substrates. Most preferred is the use of de-ionized water.

Brief Summary Text (54):
Preferably, the glycol ether component of the mixture contains an effective amount of the glycol ether material of the formula: R.sub.11 --O--R.sub.12, where R.sub.11 may be substituted by R.sub.10 --O--, where R.sub.10 can be C.sub.2 -C.sub.20 alkyl, C.sub.5 -C.sub.6 cycloalkyl, C.sub.1 -C.sub.6 glycol ether acetate, benzyl, furanyl or tetrahydrofuranyl, R.sub.11 is C.sub.1 -C.sub.20 alkyl, C.sub.5 -C.sub.6 cycloalkyl, benzyl, phenyl, furanyl or tetrahydrofuranyl, R.sub.12 is hydrogen or an alcohol selected from claim 7 above. Examples of these glycol ethers are ethylene glycol methyl ether, diethylene glycol methyl ether, ethylene glycol ethyl ether, diethylene glycol ethyl ether, ethylene glycol propyl ether, diethylene glycol propyl ether, ethylene glycol butyl ether, diethylene glycol butyl ether, propylene glycol methyl ether, propylene glycol acetate, dipropylene glycol, dipropylene glycol methyl ether, dipropylene glycol methyl ether acetate, propylene glycol propyl ether, dipropylene glycol propyl ether, propylene glycol butyl ether, and dipropylene glycol butyl ether. R.sub.10, R.sub.11 and R.sub.12 can be a C.sub.1 to C.sub.10 alkyl, preferably C.sub.1 to C.sub.6 alkyl, more preferably C.sub.1 to C.sub.4 alkyl. Among the most preferred are propylene glycol butyl ether, dipropylene glycol methyl ether, dipropylene glycol methyl ether acetate, dipropylene glycol, and diethylene glycol butyl ether.

Brief Summary Text (55):
Preferably, the pyrrolidone component of the mixture contains an effective amount of the pyrrolidone material that is substituted in the N position of the pyrrolidone ring of the formula by hydrogen, C.sub.1 to C.sub.6 alkyl, or C.sub.1 to C.sub.6 alkanol. Examples of these pyrrolidones are pyrrolidone, N-methyl pyrrolidone, N-ethyl pyrrolidone, N-propyl pyrrolidone, N-hydroxymethyl pyrrolidone, N-hydroxyethyl pyrrolidone, and N-hexyl pyrrolidone. Among the most preferred are N-methyl pyrrolidone and N-ethyl pyrrolidone.

Brief Summary Text (57):
The content of the additional components in the mixture of the present invention is not particularly critical, but for the addition of an effective amount necessary to improve or control solubility, volatility, boiling point, flammability, surface tension, viscosity, reactivity, and material compatibility. The mixture may also be enhanced by one skilled at the art by the addition of buffering agents, surfactants, chelating agents, colorants, dyes, fragrances, indicators, inhibitors, and other ingredients, all of which are well-known to those skilled in the art.

Brief Summary Text (58):
Any compound or mixture of compounds suitable for reducing the pH of the cleaner solutions of this invention, and which do not unduly adversely inhibit the cleaning action thereof or interfere with the resulting cleaned parts, may be employed. As examples of such compounds are, for example, acids, bases and their salts acting as buffers, such as inorganic mineral acids and their salts, weak organic acids having a pKa of greater than 2 and their salts, ammonium salts, and buffer systems such as weak acids and their conjugate bases, for example, acetic acid and ammonium acetate. Preferred for use as such components are acetic acid, boric acid, citric acid potassium biphthalate, mixtures of ammonium chloride and ammonium acetate, especially a 1:1 mixture of these two salts, and mixtures of acetic acid and ammonia and other amines.

Detailed Description Text (3):
An electronic hybrid microcircuit was selected that has been contaminated with an RA type flux, along with common residual oils, greases and salts common to the electronic assembly manufacturing process. The contaminated part was immersed in a solution of 97% tetrahydrofurfuryl alcohol, 1% 3 methoxy-3-methyl-1-butanol, 0.9% monoethanolamine, and 1.1 surfactants and inhibitors at 150 to 160.degree. F. (ca. 650 to ca. 71.degree. C.) for 10 minutes. The part was removed from the solution, rinsed with water and allowed to air dry. Upon visual inspection the contaminants were observed to be removed. Upon further inspection it appears the formulation removed a polyurethane coating from a wire on the part, which was not a desired material to remove from the part.

Detailed Description Text (5):
An electronic hybrid microcircuit the same as that used in Example 1 was selected that has been contaminated with an RA type flux, along with common residual oils, greases and salts common to the electronic assembly manufacturing process. The contaminated part was immersed in a solution of 1% tetrahydrofurfuryl alcohol, 97% 3 methoxy-3-methyl-1-butanol, 0.9% monoethanolamine, and 1.1% surfactants and inhibitors at 150 to 160.degree. F. (ca 65.degree. to ca. 71.degree. C.) for 10 minutes. The part was removed from the solution, rinsed with water and allowed to air dry. Upon visual inspection the contaminants were observed to be removed. In addition the urethane coating seemed to be intact with no visual signs of removal or damage.

Detailed Description Text (8):
further contaminated with fingerprint oils and dirt. The contaminated part was immersed in a solution of 35% tetrahydrofurfuryl alcohol, 35% 3 methoxy-3-methyl-1-butanol, and 30% dipropylene glycol monomethyl ether acetate at 120.degree. F. (ca. 50.degree. C.) for 3 minutes. The part was removed from the solution, rinsed with water and allowed to air dry. Upon visual inspection the contaminants were observed to be removed.

Detailed Description Text (10):
A conductive ink contaminated with a number of contaminants typical to the manufacture of capacitors and resistors was hardened on the external side of a steel test coupon and the coupon was further contaminated with fingerprint oils and dirt. The contaminated part was immersed in a solution of 35% tetrahydrofurfuryl alcohol, 35% 3 methoxy-3-methyl-1-butanol, and 30% dipropylene glycol monomethyl ether acetate at 130.degree. F. (ca. 55.degree. C.) for 1 minute. The part was removed from the solution, rinsed with water and allowed to air dry. Upon visual inspection the contaminants were observed to be removed.

Detailed Description Text (12):
A ceramic slip material used to make capacitors and resistors, contaminated with a number of contaminants typical to the manufacture of capacitors and resistors, was hardened on the external side of a steel test coupon and the coupon was further contaminated with fingerprint oils and dirt. The contaminated part was immersed in a solution of 35% tetrahydrofurfuryl alcohol, 35% 3-methoxy-3-methyl-1-butanol, and 30% dipropylene glycol monomethyl ether acetate at 135.degree. F. (ca. 60.degree. C.) for 8 minutes. The part was removed from the solution, rinsed with water and allowed to air dry. Upon visual inspection the contaminants were observed to be

removed.

Detailed Description Text (14):
An electronic circuit board was selected that has been contaminated with three types of flux, RA, RMA and a low solids "No-Clean" flux, along with common residual oils, greases and salts common to the electronic assembly manufacturing process. The contaminated part was spray washed using an inline cleaning machine having a cleaning solution of 0.7% tetrahydrofurfuryl alcohol, 18% 3 methoxy-3-methyl-1-butanol, 1.9% monoethanolamine, and 1.1% surfactants and inhibitors and 79.4% water at 150 to 160.degree. F. (ca 65.degree. to ca. 71.degree. C.) for 3 minutes in the wash section, 2 minutes in the water rinse section. The board was moved by conveyor through the wash and dry sections and was dried in a heated dryer section. Upon visual inspection the contaminants were observed to be completely removed, with the exception of some white residue remaining from resin like substances in the no-clean flux.

Detailed Description Text (16):
A photoresist polymer contaminated with a number of contaminants typical to the manufacture of semiconductors and rosin flux residue was selected. The photoresist was hardened on the external side of a silicon wafer via a baking process common to wafer manufacturing and the wafer was further contaminated with fingerprint oils and dirt. The contaminated part was immersed in a solution of 35% tetrahydrofurfuryl alcohol, 30% 3 methoxy-3-methyl-1-butanol, and 15% amino methyl propanol, 5% hexamethyldiamine and 15% water at 185.degree. F. (ca. 85.degree. C.) for 10 minutes. The part was removed from the solution, rinsed with water and allowed to air dry. Upon visual inspection the contaminants were observed to be removed.

Detailed Description Text (18):
A number of contaminants typical to many manufacturing processes were was selected. The selected contaminants were: motor oil, bearing grease, lipstick, adhesive, epoxy coating, latex paint, beeswax, RA flux, and low solids no clean flux. Steel test coupons were contaminated with the soils and allowed 24 hours to dry, bake or cure, the coupon was further contaminated with fingerprint oils and dirt in sample preparation process. The contaminated part was immersed in a solution of 1% tetrahydrofurfuryl alcohol, 80% 3 methoxy-3-methyl-1-butanol, and 19% water at 140.degree. F. (ca. 60.degree. C.) for 2 minutes. The two minute cleaning interval was selected to easily indicate cleaning differences with the cleaning solutions and soils, although it is believed the soil can be fully cleaned given a longer cleaning time and/or with the use of mechanical energy. The part was removed from the solution, rinsed with water and allowed to air dry. The coupon was visually inspected and was graded on a scale from 1 to 5 with 1 being poor cleaning, 5 being visually cleaned. The results are listed below:

Detailed Description Text (20):
Using the methods of Examples 8-16, coupons contaminated with contaminants typical to many manufacturing processes were immersed in a solution of 10% tetrahydrofurfuryl alcohol, and 90% 3 methoxy-3-methyl-1-butanol at 140.degree. F. (ca. 60.degree. C.) for 2 minutes. The part was removed from the solution, rinsed with water and allowed to air dry. The coupon was visually inspected and was graded on a scale from 1 to 5 with 1 being poor cleaning, 5 being visually cleaned. The results are listed below:

Detailed Description Text (22):
Using the methods of Examples 8-16, coupons contaminated with contaminants typical to many manufacturing processes were immersed in a solution of 1% tetrahydrofurfuryl alcohol, 94% 3 methoxy-3 methyl-1-butanol and 5% monoethanolamine at 140.degree. F. (ca. 60.degree. C.) for 2 minutes. The part was removed from the solution, rinsed with water and allowed to air dry. The coupon was visually inspected and was graded on a scale from 1 to 5 with 1 being poor cleaning, 5 being visually cleaned. The results are listed below:

Detailed Description Text (24):
Using the methods of Examples 8-16, coupons contaminated with contaminants typical to many manufacturing processes were immersed in a solution of 1% tetrahydrofurfuryl alcohol, 94% 3 methoxy-3-methyl-1-butanol and 5% dipropylene glycol methyl ether at

140.degree. F. (ca. 60.degree. C.) for 2 minutes. The part was removed from the solution, rinsed with water and allowed to air dry. The coupon was visually inspected and was graded on a scale from 1 to 5 with 1 being poor cleaning, 5 being visually cleaned. The results are listed below:

Detailed Description Text (26):
Using the methods of Examples 8-16, coupons contaminated with contaminants typical to many manufacturing processes were immersed in a solution of 1% tetrahydrofurfuryl alcohol, 94% 3 methoxy-3-methyl-1-butanol and 5% dipropylene glycol methyl ether acetate at 140.degree. F. (ca. 60.degree. C.) for 2 minutes. The part was removed from the solution, rinsed with water and allowed to air dry. The coupon was visually inspected and was graded on a scale from 1 to 5 with 1 being poor cleaning, 5 being visually cleaned. The results are listed below:

Detailed Description Text (27):
Although the invention has been described and illustrated in detail, it is to be clearly understood that the same is by way of illustration and example, and is not to be taken as a limitation. The spirit and scope of the present invention are to be limited only by the terms of the appended claims. One skilled in the art can make many adjustments, changes, or modifications to the components of the compositions used to clean contaminants from solid surfaces without departing from the spirit or scope of this invention. For example, more than one combination of the cleaning compositions can be used sequentially to clean an article or part, optionally employing different types of methods for the composition to contact the article or part, and optionally under differing conditions.

CLAIMS:

5. The composition of claim 1, further comprising water.

6. The composition of claim 1, further comprising a surfactant.

19. A method according to claim 9, further comprising rinsing the surface with water.

20. A method according to claim 9, wherein the surface is modified by a surfactant.

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DOCUMENT-IDENTIFIER: US 5728668 A
TITLE: Cleaning composition

Abstract Text (1):
An pre-spotting composition which comprises by weight percent of about 0.1 to about 15 wt. % of [(R).sub.3 N.sup.+ R'(OH).sup.- ] wherein R is a methyl or ethyl group, R' is an alkyl or ethoxylated alkyl group having about 10 to about 20 carbon atoms and water and optionally an alkali metal silicate, a cyclic nitrogen containing compound; an alknolamine; and a surfactant.

Brief Summary Text (2):
This invention relates to aqueous cleaning compositions in the form of liquids, sprays, gels and pastes, which remove dried-on and cooked-on food and other difficult-to-remove soils from kitchen utensils, flatware, dishes, glassware, cookware, bakeware, cooking surfaces, and surrounding areas in a convenient, easy, timely, and mild manner.

Brief Summary Text (7):
Deficiencies in these cleaning methods include time consumption for soaking and scouring, physical effort required for scouring and scraping, irritation to hands from harsh cleaning chemicals and methods, damage to objects from harsh chemicals and methods, unpleasant fumes and odors, and danger from heated solutions. Though non-caustic cleaners are listed in the literature, none are directed to the aqueous cleaning compositions of the present invention.

Brief Summary Text (9):
The pre-spotting aqueous cleaning compositions of the present invention comprises a tetraalkyl ammonium hydroxide and water and optionally an alkali metal silicate, a cyclic nitrogen containing compound, an alkanolamine, and a surfactant. These compositions may be formulated as clear, single-phase aqueous liquids, gels, or pastes and dispensed from bottles, squeeze bottles, or paste dispensers. It has been found that applying the caustic-free aqueous compositions of the present invention to soiled surfaces removes the above mentioned soils at ambient temperature in a relatively short period of time (from 10 to 60 minutes) without need for heat, long soaking times, scouring, or harsh chemicals.

Brief Summary Text (11):
However, it has been discovered that polymerized fats contain ester bonds which are susceptible to hydrolysis by hydroxide ion. Molecules of the type [(R).sub.3 N.sup.+ (R')X.sup.- ] (R, R'=alkyl, X=halide) have been used in organic synthesis as phase transfer catalysts because they have the ability to carry water insoluble anions such as OH.sup.- into nonaqueous solution. This permits delivery of OH.sup.- to the hydrophobic surface of baked-on fat soils.

Brief Summary Text (12):
The [(R).sub.3 N.sup.+ (R')OH.sup.- ], R.dbd.CH.sub.3, R'=tallow is prepared by ion exchange of the corresponding Cl.sup.- salt using amberlite IRA-400 (OH) ion exchange resin in either methanol or water. Preparation in methanol yields the hydroxide salt completely free of chloride but a small amount of methanol remains in the sample.

Brief Summary Text (15):

The aqueous oven cleaning compositions according to the present invention comprise approximately by weight percent:

Brief Summary Text (17):
(b) 0 to 15% of a surfactant selected from the group consisting of anionic surfactants and nonionic surfactants and mixtures thereof;

Brief Summary Text (21):
(f) 0 to 6 wt. %, more preferably 0.1 to 6 wt. % of a water soluble cosolvent such as diethylene glycol monobutyl ether and ethylene glycol monobutyl ether; and

Brief Summary Text (22):
(g) the balance being water, wherein the aqueous composition does not contain any organosilane compound containing a hydrolyzable group, any organic quaternary ammonium chloride compounds, any halogenated hydrocarbon solvents, and any polyglycol such as polyethylene glycol having a molecular weight of 200 to 600.

Brief Summary Text (26):
The pre-spotting compositions of this invention are comprised of the following components: a tetraalkyl ammonium hydroxide having the formula [(R).sub.3 N.sup.+ R'(OH).sup.- ] wherein R is an ethyl or methyl group and R' is an alkyl group having about 10 to 20 carbon atoms, preferably about 10 to 16 carbon atoms such as a coco, tallow, cetyl, lauryl or ethoxylated group, optionally, a surfactant, optionally, an alkanolamine, optionally, an imidazole, water, optionally, a solvent, and optionally a builder. In addition to the above ingredients, the compositions of this invention may contain other substances generally present in detergent compositions. Foam stabilizing agents may be incorporated at a concentration of 0.1 to 5 wt. %, and other ingredients which may normally be present at a concentration of 0.1 to 5 wt. % include preservatives, humectants, foam boosters, anti-foaming agents, dispersants, pH modifiers, colorants, and perfumes.

Brief Summary Text (27):
The surfactant, which is present in the composition in the amount of about 0 to about 15 wt. %, more preferably 0.1 to 10 weight %, is selected from the group consisting of nonionic surfactants anionic surfactants and mixtures thereof. Preferably, the surfactant is present in the amount of 1 to 7 wt. %.

Brief Summary Text (28):
The nonionic surfactant, preferably, is comprised of one or a mixture of primary alcohol ethoxylates or secondary alcohol ethoxylates or alkyl phenol ethoxylates. The primary alcohol ethoxylates are represented by the general formula:

Brief Summary Text (29):
wherein R is an alkyl radical having from 9 to 16 carbon atoms and the number of ethoxylate groups, n, is from 5 to 12. Commercially available nonionic surfactants of this type are sold by Shell Chemical Company under the tradename Neodol and by Union Carbide Corporation under the tradename Tergitol.

Brief Summary Text (31):
Wherein x+y is from 6 to 15 and the number of ethoxylate groups, n, is from 5 to 12. Commercially available surfactants of this type are sold by Union Carbide Corporation under the tradename Tergitol S series surfactants, with Tergitol 15-S-9 (T 15-S-9) being preferred for use herein.

Brief Summary Text (33):
where the number of ethoxylate groups, n, is from 8 to 15, and R is an alkyl radical having 8 or 9 carbon atoms. Commercially available nonionic surfactants of this type are sold by Rohm and Haas Company under the tradenames Triton N and Triton X series.

Brief Summary Text (34):
The anionic surfactant is preferably selected from the group consisting of paraffin sulfonate, alkyl sulfate, alkyl ethoxy sulfate, or alkyl benzene sulfonates, such as sodium linear tridecyl or dodecyl benzene sulfonate, sodium and/or ammonium alcohol ethoxy sulfate (AEOS), sodium lauroyl, cocoyl or myristoyl sarcosinate or a

combination thereof.

Brief Summary Text (35):
Alkylpolysaccharides surfactants which are also useful alone or in conjunction with the aforementioned surfactants and have a hydrophobic group containing from about 8 to about 20 carbon atoms, preferably from about 10 to about 16 carbon atoms, most preferably from 12 to 14 carbon atoms, and polysaccharide hydrophilic group containing from about 1.5 to about 10, preferably from 1.5 to 4, and most preferably from 1.6 to 2.7 saccharide units (e.g., galactoside, glucoside, fructoside, glucosyl, fructosyl, and/or galactosyl units). Mixtures of saccharide moieties may be used in the alkylpolysaccharide surfactants. The number x indicates the number of saccharide units in a particular alkylpolysaccharide surfactant. For a particular alkylpolysaccharide molecule x can only assume integral values. Any physical sample can be characterized by the average value of x and this average value can assume non-integral values. In this specification the values of x are to be understood to be average values. The hydrophobic group (R) can be attached at the 2-, 3-, or 4-positions rather than at the 1-position, (thus giving e.g. a glucosyl or galactosyl as opposed to a glucoside or galactoside). However, attachment through the 1-position, i.e., glucosides, galactosides, fructosides, etc., is preferred. In the preferred product the additional saccharide units are predominately attached to the previous saccharide unit's 2-position. Attachment through the 3-, 4-, and 6-positions can also occur. Optionally and less desirably there can be a polyalkoxide chain joining the hydrophobic moiety (R) and the polysaccharide chain. The preferred alkoxide moiety is ethoxide.

Brief Summary Text (38):
The alkyl monosaccharides are relatively less soluble in water than the higher alkylpolysaccharides. When used in admixture with alkylpolysaccharides, the alkylmonosaccharides are solubilized to some extent. The use of alkylmonosaccharides in admixture with alkylpolysaccharides is a preferred mode of carrying out the invention. Suitable mixtures include coconut alkyl, di-, tri-, tetra-, and pentaglucosides and tallow alkyl tetra-, penta-, and hexaglucosides.

Brief Summary Text (41):
The amount of unreacted alcohol (the free fatty alcohol content) in the desired alkylpolysaccharide surfactant is preferably less than about 2%, more preferably less than about 0.5% by weight of the total of the alkylpolysaccharide. For some uses it is desirable to have the alkylmonosaccharide content less than about 10%.

Brief Summary Text (42):
The used herein, "alkylpolysaccharide surfactant" is intended to represent both the preferred glucose and galactose derived surfactants and the less preferred alkylpolysaccharide surfactants. Throughout this specification, "alkylpolyglucoside" is used to include alkyl- polyglycosides because the stereo chemistry of the saccharide moiety is changed during the preparation reaction.

Brief Summary Text (43):
An especially preferred APG glycoside surfactant is APG 625 glycoside manufactured by the Henkel Corporation of Ambler, Pa. APG 25 is a nonionic alkylpolyglycoside characterized by the formula:

Brief Summary Text (44):
wherein n=10(2%); n=12(65%); n=14(21-28%); n=16(4-8%) and n=18(0.5%) and x(degree of polymerization)=1.6. APG 625 has: a pH of 6-8(10% of APG 625 in distilled water); a specific gravity at 25.degree. C. of 1.1 grams/ml; a density at 25.degree. C. of 9.1 kgs/gallons; a calculated HLB of about 12.1 and a Brookfield viscosity at 35.degree. C., 21 spindle, 5-10 RPM of about 3,000 to about 7,000 cps. Mixtures of two or more of the liquid nonionic surfactants can be used and in some cases advantages can be obtained by the use of such mixtures.

Brief Summary Text (46):
The composition contains about 0 to 25 wt. %, more preferably 0.1 to 10 wt. %, of a penetrant which is a cyclic nitrogen containing compound such as pyrrolidine, pyridine, 2-pyrrolidine, N-methyl, 2-pyrrolidone, imidazole, morpholine, and diethylenetriamine, wherein the preferred cyclic nitrogen containing compounds are

imidazole and 4-methylimidazone. Imidazole is especially preferred. Triethylenediamine or 1,4-diaza bicylco [2,2,2] octane also works as a penetrant in the instant composition.

Brief Summary Text (48):
The alkali metal silicate can be optionally present in the composition at a concentration of 0 to 50 wt. %, more preferably about 5 to about 45 wt. % and most preferably about 10 to about 40 wt. %. The alkali metal silicates are preferably sodium silicate and/or potassium silicate. The potassium silicate is characterized by the formula K.sub.2 OXSiO.sub.2 wherein x>2.10 and the potassium silicate has a water content of less than 66 wt. %. The sodium silicate is characterized by the formula Na.sub.2 OXSiO.sub.2 wherein x>2.88 and the sodium silicate has a water content of less than 61 wt. %. Water completes the balance of the composition and the pH of the composition is about 11 to about 13.

Brief Summary Text (49):
The compositions of this invention are prepared by adding with stirring in a suitable mixer and homogenizer at a temperature of about 15.degree. C. to about 30.degree. C. an aqueous solution of the tetraalkyl ammonium hydroxide, imidazole and/or alkanolamine to an aqueous solution of a surfactant and alkali metal silicate selected from the group consisting of lithium silicate, sodium silicate and potassium silicate, wherein the alkali metal silicate is in an aqueous solution at a concentration of about 30 wt. % to about 60 wt. %.

Brief Summary Text (51):
wherein x=1, 2 or 3 and n is about 2 to about 8 and the concentration of the organic compound is about 0 to about 15 wt. %, 0.1 to about 15 wt. %. Also suitable as structuring agents are nonionic surfactants containing a hydroxyl group.

Detailed Description Text (4):
The solutions were prepared by simple mixing at R.T. the water and the tetraalkyl ammonium hydroxide or chloride.

Detailed Description Text (5):
These formulations were tested on Crisco shortening baked at 350.degree. F. for 10 hours on a 2 inch pyrex petri dishes. Approximately 4 g of each formula was used and the soil was soaked at room temperature for 16 hours. The formulations were then rinsed off with tap water and light rubbing to remove loosened soil. Percent soil removal was determined gravimetrically. Three replicates were run for each formula.

Detailed Description Text (8):
These formulations were tested on 80% lean ground beef baked at 350.degree. F. for 45 minutes on 2 inch pyrex petri dishes and 2 inch stainless steel planchettes. Approximately 6 g of ground beef was placed on each substrate. After baking, each patty was removed and the substrates were then weighed before and after cleaning. Approximately 2 g of each formula was used and the soil was soaked at room temperature for 5 minutes. The formulations were then rinsed off with with 80.degree.-90.degree. F. tap water. Percent soil removal was determined gravimetrically. Three replicates were run for each formula. Two commercial formulations were also tested: Fantastic All Purpose Cleaner was used as purchased and a solution of 1% Palmolive Dishwashing Liquid was diluted to 1% with tap water was also tested. Results are shown below.

Detailed Description Paragraph Table (1):
TABLE I

| | A | B | C |
|---|---|---|---|
| (R).sub.3 N.sup.+ (R')OH.sup.-, 0.5 1.0 0 R = CH.sub.3 ; R' = tallow | | | |
| Water | 99.5 | 99.0 | |
| R.sub.3 N.sup.+ (R')Cl.sup.-, 0 0 1.0 R = CH.sub.3 ; R' = tallow | | | |
| pH | 12.2 | 12.6 | 12.2 |
| % soil removal | 69 | 89 | 12 |

Detailed Description Paragraph Table (2):

| | D | E |
|---|---|---|
| (R).sub.3 N.sup.+ (R')OH.sup.- 1.0 0.0 R = CH.sub.3 ; R' = C.sub.14 H.sub.29 | | |
| (R).sub.3 N.sup.+ (R')OH.sup.- 0.0 1.0 R = CH.sub.3 ; R' = tallow | | |
| NaLAS | 2.0 | 2.0 |
| Diethyleneglycol Monobutyl Ether | 5.0 | 5.0 |
| Perfume | 0.2 | 0.2 |
| Deionized water | bal. | bal. |

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CLAIMS:

1. A baked-on soil prespotting aqueous cleaning composition which consists of by weight percent:

(a) 5 to about 45 of an alkali metal silicate;

(b) 0.1 to about 10 of a cyclic nitrogen containing compound,

(c) 0.1 to about 0.6 of an alkanolamine;

(d) 0.1 to 10 of at least one anionic surfactant;

(e) 0.1 to about 15.0 of (R.sub.3)N.sup.+ R'(OH.sup.-), wherein R is methyl or ethyl group and R' is an alkyl or ethoxylated alkyl group having about 10 to about 20 carbon atoms;

(f) 0.1 to 15 of an organic compound of the formula C.sub.n H.sub.2n+2-x (OH).sub.x wherein x=1, 2 or 3 and n is 2 to 8; and

(g) the balance being water.

L12: Entry 61 of 90 File: USPT Aug 23, 1994

DOCUMENT-IDENTIFIER: US 5340488 A
TITLE: Composition for cleaning an internal combustion engine

Abstract Text (1):
A composition is disclosed which cleans baked-on, carbonized sludges and varnish deposits from the internal surfaces of automobile, motorcycle and truck engines. The product comprises a mutually synergistic solution of selected cyclic compounds, aliphatic amines and water, preferably dissolved in a hydrocarbon or hydrocarbon and aliphatic lower alcohol fuel base. The solution or base product may be placed in a pressure resistant (aerosol) dispenser, and pressurized. When injected into an engine over a period of time, significant removal of deposited contaminants occurs, resulting in smooth and efficient engine operation and lower levels of unburned hydrocarbon fuel, carbon monoxide, and other air pollutants.

Drawing Description Text (2):
FIG. 1 Cleaning efficiency of N-methyl-2-pyrrolidone and n.butylamine blends on spark plug sludge in a five (5) minute test. The dashed line represents the theoretical (expected) curve. The solid line represents the actual (synergistic) curve.

Drawing Description Text (7):
20% N-methyl-2-pyrrolidone

Drawing Description Text (18):
21% N-methyl-2-pyrrolidone

Detailed Description Text (2):
The subject invention provides a composition for dissolving organic matter which comprises (i) a hydrazine or an aliphatic amine, and (ii) a hydrocarbon or substituted hydrocarbon compound having a ring wherein at least one-half the atoms composing the ring are carbon. The compound and the hydrazinc or amine are compatible with each other and are capable of forming a homogeneous mixture. Typically, this compound comprises (i) a hydrazine (e.g. pure hydrazine, hydrated hydrazine or methylated hydrazine) or an aliphatic amine, and (ii) a ring-containing compound wherein the ring is composed of three, five or six atoms consecutively linked. The term "compatible" as used throughout the application is to include miscible, partially miscible, or immiscible components which can be made at least partially miscible by the use of an agent such as a surfactant, enzyme, catalyst, co-solvent, etc.

Detailed Description Text (6):
A multitude of two-component blends containing a hydrazine (e.g. pure, hydrated or methylated) and/or aliphatic amine and a ring containing hydrocarbon or substituted hydrocarbon compound have been tested. Many of these blends exhibit synergistic activity, allowing the removal of varnish/sludge deposits on spark plugs with outstanding speed and efficiency. In the case of the synergistic compositions, removal values for specific blends were substantially greater than would have been predicted from the removal values of the individual solvents. For example, a blend of 75% N-methyl-2-pyrrolidone and 25% n.butylamine showed 97 per cent removal of light, baked-on varnish/sludge deposits. However, only a 26 per cent removal would be predicted based upon a weighted average of the removal ratings of the pure components.

Detailed Description Text (9):
Many of the above compounds are strong solvents. However, none individually were effective against burned-on varnishes. Each produced strong synergism with hydrazines and/or amines, at 5-15% amine or higher for the heterocyclics, and 20% or higher for the toluene/n.butylamine blend. Various combinations of a ring containing compound and a hydrazine and/or an aliphatic amine were found to be effective in removing coke and burned on sludge. For example, 75:25 weight percent mixtures of ring containing compound to hydrazine monohydrate showed excellent removal when the ring containing compound was N-methyl-2-pyrrolidone, 2-pyrrolidone-1-hydrate, 1-vinyl-2-pyrrolidone, furfurylamine and furfuryl alcohol.

Detailed Description Text (10):
N-methyl-2-pyrrolidone and n.butylamine demonstrated high varnish/sludge removal efficiency (to 97 per cent), and N-methyl-2-pyrrolidone and isopropylamine was almost as high (to 85 per cent) and was later found to be at least as effective as the N-methyl-2-pyrrolidone and n.butylamine couple when diluted into the finished concentrate.

Detailed Description Text (11):
In a preferred embodiment of the sueject invention, the composition for dissolving organic matter further comprises an amount of water effective to increase the dissolving of varnish and burned-on sludge. Most preferably, the water comprises less than about 4.0% of the composition when employing alkylamines, and less than about 15% when using hydrazine.

Detailed Description Text (12):
In order to gain the benefit of additional water, a simple co-solvent may be employed. Such co-solvents include methanol, ethanol, isopropanol and n.propanol. Isopropanol is preferred because it is non-poisonous, available without denaturants or payment of special taxes and is commercially available. At about 10 per cent or so of the final formula, isopropanol at least doubles the amount of water which may be incorporated into the concentrate without phase separation. It is also thought to aid in combustion (e.g. "gasohol" type blends are predominantly gasolines and ethanol).

Detailed Description Text (14):
It is preferred that the composition comprises an engine fuel. In optimally applying the teaching of the invention, a synergistic pair of cyclic and amine compounds is prepared, and to this is added about 50 per cent or more of iso-octanes or similar engine fuel. About 10 per cent of isopropanol or similar simple alcohol may then be added. Deionized water or aqueous ammonia may be added in an amount of about 1 to 4 per cent as the third member of synergistic varnish/sludge remover triad. This is best accomplished by completing a concentrate batch except for water addition, then removing about 10 per cent into a separate vessel. The main batch is then titrated with successive amounts of water or aqueous ammonia until incipient phase separation occurs, as evident by the liquid becoming hazy. After several minutes of further stirring, the haziness dissipates. At that point, the separated 10 per cent portion is added back in and the batch is complete.

Detailed Description Text (17):
It is particularly preferred that the amine is selected from the group consisting of n-butylamine, triethylamine, diethylamine, and isopropylamine; the hydrazine is selected from the group consisting of hydrazine, monomethyl hydrazine and hydrazine monohydrate; and the ring-containing compound is selected from the group consisting of N-methyl-2-pyrrolidone, furfurylamine 2-pyrrolidone hydrate, N-vinyl-2-pyrrolidone, furfuryl alcohol, 2-methylaziridine, tetramethylene sulfone, 2-methylimidazole, 3-methyl-2-oxazolidinone, 2-methyl-2-oxazoline, tetrahydrofuran, 4-butyrolactone, toluene, and xylene. Normally, the weight ratio of the ring-containing compound to the aliphatic amine is from 3:96 to 96:3.

Detailed Description Text (18):
Synergistic pairs of cyclic and simple amines and/or hydrazines also work fairly well without the need for added water or aqueous ammonia.

Detailed Description Text (20):
The synergistic effect of the N-methyl-2 -pyrrolidone/n.butylamine couple and the N-methyl-2-pyrrolidone/diethylamine couple is presented in FIGS. 1 and 2.

Detailed Description Text (22):
The following two formulae illustrate the value of added water in improving cleaning efficacy.

Detailed Description Text (24):
In another embodiment, the composition of matter further comprises anhydrous ammonia or ammonium hydroxide, more preferably in combination with triethylamine. Most preferably, the triethylamine is present in a concentration of greater than about 10%. Typically, this mixture comprises about 25 per cent of the formula. Since it is necessary to have the engine running during the injection and cleaning phase (approximately five minutes), a suitable fuel is included in all finished formulations. Various gasolines may be used, but for improved performance and better product uniformity, the use of a specific commercial mixture of predominantly isomeric isooctanes is recommended. The final ingredient is a simple aliphatic alcohol, used as a co-solvent to enable the inclusion of a higher level of water before phase separation occurs. Up to about ten per cent of other ingredients may be included as lubricants or for other non-essential purposes.

Detailed Description Text (25):
The subject invention provides in a most preferred embodiment, a three-component, mutually synergistic blend, having a high pH value (typically about 13.0) which, when dissolved in a suitable fuel base, may be injected into engines to perform a cleaning function that is dramatically superior to that of any known product. One composition demonstrating three-way synergism without water or aqueous ammonia comprises isopropylamine, tetrahydrofuran and hydrazine monohydrate in a 2:1:1 ratio.

Detailed Description Text (30):
One preferred dispenser comprises about 35.0% by weight of N-methyl-2-pyrrolidone; 15.0% by weight of diethylamine; 49.0% by weight of iso-C.sub.7-8 -paraffinics; 0.5% by weight of deionized water; and 0.5% by weight of nitrogen gas.

Detailed Description Text (31):
Also provided for is a dispenser which comprises about 35.0% by weight of N-methyl-2-pyrrolidone; 15.0% by weight of isopropylamine; 48.5% by weight of iso-C.sub.7-8 -paraffinics; 1.1% by weight of deionized water; and 0.4% by weight of nitrogen gas.

Detailed Description Text (32):
The dispenser may also comprise about 30.0% by weight of tetrahydrofuran; 10.0% by weight of n.butylamine; 49.0% by weight of iso-C.sub.7-8 -paraffinics; 0.5% by weight of deionized water; 10.0% by weight of methyl t.butylether; and 0.5% by weight of nitrogen gas.

Detailed Description Text (33):
Yet another dispenser comprises about 25.0% by weight of 4-butyrolactone; 12.0% by weight of n.pentylamine; 41.0% by weight of unleaded gasoline; 1.0% by weight of deionized water; 10.0% by weight of isopropanol (anhydrous); 10.0% by weight of methyl t.butylether; 0.5% by weight of lubricating oil; and 0.5% by weight of nitrogen gas.

Detailed Description Text (34):
Another dispenser comprises about 18.0% by weight of toluene; 22.0% by weight of n.butylamine; 10.0% by weight of unleaded gasoline; 44.3% by weight of n.C.sub.9-10 -paraffinics; 1.2% by weight of deionized water; 3.9% by weight of methanol (99.5%); and 0.6% by weight of nitrogen gas.

Detailed Description Text (35):
A further dispenser comprises about 30.0% by weight of hydrazine monohydrate; 15.0% by weight of N-methyl-2-pyrrolidone; 42.5% by weight of iso-C.sub.7-8 -paraffinics; 12.0% by weight of ethanol (anhydrous); and 0.5% by weight of nitrogen gas.

Detailed Description Text (36):
Another preferred dispenser comprises about 16.0% by weight of mixed isometric xylenes; 24.0% by weight of diethylamine; 49.0% by weight of unleaded gasoline; 0.8% by weight of deionized water; 9.7% by weight of n.propanol; and 0.5% by weight of nitrogen gas.

Detailed Description Text (37):
A further dispenser comprises about 5.0% by weight of N-methyl-2-pyrrolidone; 10.0% by weight of triethylamine; 18.0% by weight of isopropanol (anhydrous); 4.0% by weight of xylene-based proprietary lubricant; 10.0% by weight of toluene or xylenes; 49.3% by weight of iso-(C.sub.7-8)-isoparaffinics; 3.0% by weight of ammonium hydroxide (28%); and 0.7% by weight of nitrogen gas.

Detailed Description Text (38):
Finally, a dispenser which comprises about 10.0% by weight of N-methyl-2-pyrrolidone; 22.2% by weight of 2-pyrrolidone-1-hydrate; 30.0% by weight of xylenes or toluene; 4.0% by weight of isopropanol (anhydrous); 15.0% by weight of methyl t.butylether; 2.0% by weight of ammonium hydroxide (28%); 1.7% by weight of deionized water; 0.5% by weight of Dowfax 2A1 surfactant; 14.0% by weight of triethylamine; and 0.6% by weight of nitrogen gas is provided for.

Detailed Description Text (43):
An automotive engine injector type cleaner having the composition:

Detailed Description Text (44):
was prepared by mixing the first three ingredients and then adding water to the point of incipient phase separation. After that the concentrate was packaged and the dispenser gassed with nitrogen propellant. The pH value of the concentrate was 12.5 at 25.degree. C. Removal of varnish/sludge spark plug contaminants by the CSPIT test was 55% for heavy soils, 98% for light soils and 40% for carbonized soils.

Detailed Description Text (46):
An automotive engine injector type cleaner having the composition:

Detailed Description Text (47):
was prepared as above. The pH value of the concentrate was 12.8 at 25.degree. C. before adding the water, and 13.4 at 25.degree. C. after adding the water. Removal of varnish/sludge spark plug contaminants by the CSPIT test was 55% for heavy soils, 98% for light soils and 60% for carbonized soils. This formula was higher in pH and judged more effective in soil removal than Example 1.

Detailed Description Text (49):
An automotive engine injector type cleaner having the composition:

Detailed Description Text (50):
can be prepared by mixing the ingredients, except water and nitrogen, then adding the water to the point of incipient phase separation. Package and gas with nitrogen propellant. The pH value of the concentrate is 12.6 at 25.degree. C.

Detailed Description Text (53):
can be prepared by combining all the ingredients except water and nitrogen gas, then adding the required amount of water. Package and gas with nitrogen propellant.

Detailed Description Text (55):
can be prepared by combining all the ingredients, but adding the water last; then gassing with the nitrogen propellant.

Detailed Description Text (60):
An automotive injector type cleaner having the composition:

Detailed Description Text (61):
can be prepared by combining all the ingredients except water and nitrogen, then adding the water and gassing with nitrogen propellant.

Detailed Description Text (63):
An automotive engine injector type cleaner having the composition:

Detailed Description Text (65):
This formula is typical of those found to be satisfactory with the recently introduced fluorosilicone automotive gaskets, as well as all others. The compatibility with the fluorosilicone elastomers is based on the use of not more than 10% N-methyl-2-pyrrolidone, and the use of a tertiary aliphatic amine, instead of primary and/or secondary simple aliphatic amines. The water content of this formula is 2.16%. The water is rendered soluble by the use of 18.0% isopropanol, in this case, as a co-solvent.

Detailed Description Text (67):
An automotive engine injector type cleaner having the composition:

Detailed Description Text (71):
Automobile engine cleaners having the composition:

Detailed Description Text (74):
An automotive engine injector/valve type cleaner having the composition:

Detailed Description Text (79):
The strong synergism in the N-Methyl-2-pyrrolidone/n.Butylamine system is shown by Formulae Nos. 017-020.

Detailed Description Text (83):
Strong synergism of N-Methyl-2-pyrrolidone/iso.Propylamine is shown by Formulae Nos. 038-041.

Detailed Description Text (86):
Modest synergism of Ethylene Glycol n.Butyl Ether/n.Butylamine is shown by Formulas No. 047-051.

Detailed Description Text (88):
Variable, but non-measurable amounts of water in the amine affect results significantly. The amount of water in the listed formulations is thought to be about 0.5 to 1.0%. There are two formulae identified as 066; cited here as 066 and 066+

Detailed Description Paragraph Table (1):
Ring Compound Skeletal Structure
N-Methyl-2-pyrrolidone (M-Pyrol) ##STR1## Tetrahydrofuran (THF) ##STR2## 4-Butyrolactone (gamma-Butyrolactone) ##STR3## Toluene ##STR4## 2-Pyrrolidone ##STR5## N-Vinyl-2-Pyrrolidone ##STR6## Furfurylamine ##STR7## Furfuryl Alcohol ##STR8## 2-Methylimidazole ##STR9## 3-Methyl-2-Oxazolidinone ##STR10## 2-Methyl-2-Oxazoline ##STR11## 2-Methylaziridine ##STR12## Tetramethylene Sulfone ##STR13##

Detailed Description Paragraph Table (2):
INGREDIENTS* FORMULA 72 FORMULA 73
N-Methyl-2-pyrrolidone 20% 20% n.Butylamine 4% -- Isopropylamine 4% Isopar C (Exxon, Inc.) 66% 66% Mainly iso-C.sub.7-8 -paraffinics Methyl-t.butylether 10% 10% CSPIT RESULTS Removal of light varnish/ 35% 75% sludge Removal of carbonized 20% 55% varnish/sludge
*Tramp water of Formula 69, 72 and 73 estimated as 0.3%. (None was specifically added.)

Detailed Description Paragraph Table (3):
INGREDIENTS FORMULA 69 FORMULA 82
N-Methyl-2-pyrrolidone 20.5% 20.0% n.Butylamine 5.0% 5.0% Water 0.3% 1.4% Isopar C (Exxon, Inc.) 65.2% 62.8% Mainly iso-C.sub.7-8 - paraffinics Isopropanol (Anhydrous) -- 10.8% Methyl t.butylether 9.0% -- pH Value (25.degree. C.) 12.6 13.0 CSPIT RESULTS Removal of heavy varnish/ 50% -- sludge Removal of light varnish/ 90% 93% sludge Removal of carbonized 50% 60% varnish/sludge REDUCTION OF TAILPIPE EMISSIONS** Unburned hydrocarbon fuel 81% & 81% 91%, 52% & 92% Carbon Monoxide 88% & 86% 65%, 57% & 90%

**These percentage indicate the reduction of varnish/sludge from the amounts present prior to cleaning.

Detailed Description Paragraph Table (4):
N-Methyl-2-pyrrolidone 35.0% Diethylamine 15.0% Iso-C.sub.7-8 -paraffinics 49.0% Deionzed Water 0.5% Nitrogen 0.5%

Detailed Description Paragraph Table (5):
N-Methyl-2-pyrrolidone 35.0% Isopropylamine 15.0% Iso-C.sub.7-8 -paraffinics 48.5% Deionzed Water 1.1% Nitrogen 0.4%

Detailed Description Paragraph Table (6):
Tetrahydrofuran (THF) 30.0% n.Butylamine 10.0% Iso-C.sub.7-8 -paraffinics 49.0% Deionzed Water 0.5% Methyl t.butylether 10.0% Nitrogen 0.5%

Detailed Description Paragraph Table (7):
Butyrolactone 25.0% n.Pentylamine 12.0% Unleaded Gasoline 41.0% Deionized Water 1.0% Isopropanol (Anhydrous) 10.0% Methyl t.butylether 10.0% Lubricating Oil 0.5% Nitrogen 0.5%

Detailed Description Paragraph Table (8):
Toluene 18.0% n.Butylamine 22.0% Unleaded Gasoline 10.0% n.C.sub.9-10 -paraffinics 44.3% Deionzed Water 1.2% Methanol - 99.5% 3.9% Nitrogen 0.6%

Detailed Description Paragraph Table (9):
Hydrazine Monohydrate 4.0% N-Methyl-2-pyrrolidone 10.0% Iso-C.sub.7-8 -paraffinics 63.0% Isopropanol 12.0% Toluene 10.0% Dowfax 2Al 0.5% Nitrogen 0.5%

Detailed Description Paragraph Table (10):
Mixed Isomeric Xylenes 16.0% Diethylamine 24.0% Unleaded Gasolines 49.0% Deionized Water 0.8% n.Propanol 9.7% Nitrogen 0.5%

Detailed Description Paragraph Table (11):
N-Methyl-2-pyrrolidone 5.0% Triethylamine 10.0% Isopropanol (Anhydrous) 18.0% Xylene-based lubricant 4.0% Toluene (or Xylenes) 10.0% Iso-(C.sub.7-8)-isoparaffinics 49.3% Ammonium Hydroxide (28%) 3.0% Nitrogen 0.7%

Detailed Description Paragraph Table (12):
N-Methyl-2-pyrrolidone 10.0% 2-Pyrrolidone 1-Hydrate 22.2% Xylenes or Toluene 30.0% Isopropanol (Anhydrous) 4.0% Methyl tertiary Butyl Ether 15.0% Ammonium Hydroxide (28%) 2.0% Deionized Water 1.7% Dowfax 2Al surfactant 0.5% Triethyl amine 14.0% Nitrogen 0.6%

Detailed Description Paragraph Table (13):
213A 214A
2-Pyrrolidone-1-hydrate 10.0 14.416 N-Methyl-2-pyrrolidone 12.0 9.614 Triethylamine 10.0 14.416 Isopropanol 15.5 14.600 Methyl t.butyl ether 5.8 5.188 Xylene 9.0 -- Dowfax 2AI 0.5 0.485 Ammonium hydroxide (28%) 2.2 2.881 Isopar "C" 35.0 -- Regular unleaded gasoline -- 38.400

Detailed Description Paragraph Table (14):
N-Methyl-2-pyrrolidone 8.0% Furfurylamine 2.0% Isopropanol 12.0% Toluene 10.0% Hydrazine Monohydrate 4.0% Iso-(C.sub.7 -C.sub.8)-isoparaffinics 63.0% Dowfax 2Al 0.5% Nitrogen 0.5%

Detailed Description Paragraph Table (15):

INDIVIDUAL SOLVENTS Formula Number: 001 002 003 004 005 006 007 008 009 010

Hydrazine 100 -- -- -- -- -- -- -- -- -- Diethylene Triamine -- 100 -- -- -- -- -- -- -- -- n.Butylamine -- -- 100 -- -- -- -- -- -- -- Diethylamine -- -- -- 100 -- -- -- -- -- -- Triethylamine -- -- -- -- 100 -- -- -- -- -- Tetrahydrofuran -- -- -- -- -- 100 -- -- -- -- N-Methyl-2-pyrrolidone -- -- -- -- -- -- 100 -- -- -- Butyrolactone -- -- -- -- -- -- -- -- 100 -- -- Dibutylamine -- -- -- -- -- -- -- -- -- 100 -- Ethylene Glycol n. -- -- -- -- -- -- -- -- -- -- 100 Butyl Ether (As Dowanol DB) Soil Removal 8.0 1.0 6.0 3.5 0.3 1.0 1.5 0.5 0.3 0.1 INDIVIDUAL AND BINARY SOLVENTS Formula Number: 011 012 013 014 015 016 017 018 019 020

Lubrizol 8166 100 -- -- -- -- -- -- -- -- -- -- Mixture Toluene -- 100 -- -- -- -- -- -- -- -- Methyl Ethyl Ketone -- -- 100 -- -- -- -- -- -- -- -- n.Butyl Acetate -- -- -- 100 -- -- -- -- -- -- -- -- Methylene Chloride -- -- -- -- 100 -- -- -- -- -- iso.Propylamine -- -- -- -- -- 100 -- -- -- -- N-Methyl-2- -- -- -- -- -- -- 25 50 75 90 pyrrolidone n.Butylamine -- -- -- -- -- -- 75 50 25 10 Soil Removal 0.1 0.2 0.1 0.1 0.0 5.0 5.0 9.3 9.7 9.0

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Detailed Description Paragraph Table (21):

RESIDUE REMOVAL BY FORMULATED SOLVENT BLENDS, WHEN APPLIED TO AREAS AROUND THE AUTOMOTIVE SPARK PLUG GAP FOR FIVE MINUTES AGAINST THREE TYPES OF SLUDGE DEPOSITS TESTED NO. CHEMICAL NAME % SLUDGE A SLUDGE B SLUDGE C AVG

077. N-Methyl-2- 35.0 pyrrolidone n.Butylamine 15.0 55 -- -- 55 Iso-(C.sub.7-8)- 49.84 paraffinics Deionized 0.16 Water pH = 12.8 078. N-Methyl-2- 20.5 pyrrolidone n.Butylamine 5.0 50 90 50 63 Iso-(C7-8)- 65.2 95 paraffinics Methyl t.butyl 9.0 ether Deionized 0.3 Water 079. N-Methyl-2- 35.00 pyrrolidone n.Butylamine 15.00 -- 98 60 79 Iso-(C.sub.7-8)- 48.97 55 98 50 60 Water 1.03 pH = 13.2 080. N-Methyl-2- 35.00 pyrrolidone Isopropylamine 15.00 -- 98 60 79 Iso-(C.sub.7-8)- 48.89 55 98 60 71 paraffinics Water 1.11 pH = 13.0 081. N-Methyl-2- 35.00 pyrrolidone Diethylamine 15.00 -- 90 50 70 Iso-(C.sub.7-8)- 49.50 55 98 40 64 Water 0.50 pH = 12.5 069. A.N-Methyl-2- 20.00 pyrrolidone n.Butylamine 5.0 Iso-(C7-8)- 63.0 50 93 50 63 paraffinics Isopropanol 11.7 (Anhyd.) Water 0.3 pH = 13.0 082. N-Methyl-2- 20.0 pyrrolidone n.Butylamine 5.0 -- 90 Iso-(C.sub.7-8)- 62.8 -- 95 60 76 paraffinics Isopropanol 10.8 (Anhyd.) Water 1.4 pH = 13.0

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Detailed Description Paragraph Table (22):

RESIDUE REMOVAL BY FORMULATED SOLVENT BLENDS, WHEN APPLIED TO AREAS AROUND THE AUTOMOTIVE SPARK PLUG GAP FOR FIVE MINUTES COMPARISON OF THE FORMULAE FOR THOSE BLENDS EXHIBITING AT LEAST 80% SOIL REMOVAL (AV.) Formula Number: 79 92D 94 201 207 214 215 217 221 222

N-Methyl-2 35 5 5 10 10 10 10 10 10 10 pyrrolidone 2-Pyrrolidone -- -- -- -- -- -- -- -- 25 -- 1-Hydrate Tetrahydrofuran -- -- -- -- -- -- -- -- -- -- 4-Butyrolactone -- -- -- 40 26 24 24 25 -- -- Toluene -- -- -- -- -- -- -- -- -- 10 Mixed Xylene -- -- -- 30 30 30 30 30 29 -- Isomers Aromatic 150 -- -- -- -- -- -- -- -- 10 -- Hydrazine -- -- -- -- -- -- -- -- -- 4 Monohydrate Triethylamine -- 10 10 9 14 12 10 14 20 -- Iso-(C.sub.7-8)- 49 67 64 -- -- -- -- -- -- 63.5 paraffinics 2-Nitropropane -- -- -- -- -- -- -- -- -- -- Water 1 -- -- -- -- -- -- -- -- -- Ammonium -- 3 3 1 1 2 3 2 2 -- Hydroxide (29%) Isopropanol -- 15 18 -- 4 7 8 4 4 12 Methyl tert. -- -- -- 10 15 15 15 15 -- -- butyl ether Dowfax 2A1 -- -- -- -- -- -- -- -- -- 0.5 Soil Removal 8.0 8.0 8.5 8.3 8.3 8.3 8.0 8.1 8.3 10.0 pH Value (25.degree. C.) 11.0 11.3

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Detailed Description Paragraph Table (23):

2-Pyrrolidone-1-Hydrate 14.416% N-Methyl-2-pyrrolidone 9.614% Triethylamine 14.416% Isopropanol 14.600% Methyl tert.butyl ether 5.188% Dowfax 2A1 0.485% Ammonium hydroxide 28.degree. Be' 2.881%

Regular unleaded gasoline 38.400%

CLAIMS:

1. A method of dissolving organic matter which comprises contacting the organic matter with a composition containing:

(a) a primary, secondary or tertiary aliphatic amine selected from the group consisting of primary amines having a C.sub.1-7 chain attached to the nitrogen atom, secondary amines having C.sub.1-6 chains attached to the nitrogen atom, and tertiary amines having C.sub.1-5 chains attached to the nitrogen atom; and

(b) a hydrocarbon or substituted hydrocarbon compound selected from the group consisting of N-methyl-2-pyrrolidone, 2-pyrrolidone-1-hydrate, 1-vinyl-2-pyrrolidone, furfurylamine, furfuryl alcohol, 2-methylimidazole, 3-methyl-2-oxazolidinone, 2-methyl-2-oxazoline, 2-methylaziridine, tetramethylene sulfone, tetrahydrofuran and 4-butyrolactone;

the compound and the amine being compatible with each other, capable of forming a homogeneous mixture, and present in synergistic amounts.

13. A method of claim 1, wherein the composition further comprises water in an amount effective to assist in dissolving organic matter.

14. A method of claim 13, wherein the water in the composition comprises less than 4.0% by weight of the composition.

15. A method of claim 14, wherein the water in the composition comprises less than 1.0% by weight of the composition.

22. A composition for dissolving organic matter which comprises:

(a) an aliphatic amine selected from the group consisting of n-butylamine, isopropylamine, diethylamine and triethylamine; and

(b) a hydrocarbon or substituted hydrocarbon selected from the group consisting of N-methyl-2-pyrrolidone, 2-pyrrolidone-1-hydrate, 1-vinyl-2-pyrrolidone, furfurylamine, furfuryl alcohol, 2-methylimidzole, 3-methyl-2-oxazolidinone, 2-methyl-2-oxazoline, 2-methylaziridine, tetramethylenesulfone, tetrahydrofuran, and 4-butyrolactone;

the compound and the amine being compatible with each other, capable of forming a homogeneous mixture, and present in synergistic amounts.

WEST

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L12: Entry 77 of 90 File: USPT May 12, 1987

DOCUMENT-IDENTIFIER: US 4664721 A
TITLE: Printing screen cleaning and reclaiming compositions

Abstract Text (1):
Ink cleaning compositions are disclosed which degrade or solubilize broad classes of printing ink residues on printing screens for quick removal by a pressurized water stream. The compositions are essentially non-aqueous and contain N-methyl-2-pyrrolidone (NMP), an oxygenated solvent, such as butyl cellosolve and cyclohexanone, and a surfactant. Alternatingly, compositions containing NMP and tetrahydrofurfuryl alcohol as the primary components are disclosed. Methods of cleaning and reclaiming printing screens which provide synergistic activities are also disclosed. The compositions meet health and safety standards due to their biodegradability, lack of flammability and high threshold limit values.

Brief Summary Text (3):
Commercial screen printing shops usually clean or reclaim many screens daily and, for this purpose, employ screen cleaning machines or reclaiming systems. Such cleaning machines or reclaiming systems usually employ recirculating solvents which allow the synthetic or metal screens to be introduced and either cleaned or reclaimed, depending upon production requirements. In addition, other commercial operations involve hand-cleaning or reclaiming with various solvents or corrosive agents. During the course of cleaning or reclaiming, the screen printer is often in intimate contact with the chemicals or solvents. There are many solvents or agents used in screen cleaning and reclamation. The three most commonly used agents may be classified as aliphatic hydrocarbons, aromatic hydrocarbons and oxygenated solvents, and less frequently, chlorinated solvents. Aliphatic hydrocarbons are commonly referred to as "mineral spirits" but more accurately these aliphatic solvents are composed of mixtures of straight-chain and/or branched-chain saturated hydrocarbons. The higher the molecular weight or the number of carbon atoms, the higher the boiling point of the solvent. The higher the boiling point, the slower the solvent will evaporate, hence, usually the higher boiling aliphatic hydrocarbons are desirable for use in screen cleaning. The aromatic hydrocarbon solvents include cyclic hydrocarbons containing the benzene ring. These aromatic hydrocarbons are usually more flammable but much stronger in solvation power than the aliphatic type solvents and, similarly, the higher the molecular weight for the aromatic hydrocarbon, the higher the boiling range. In contrast to the rather non-polar hydrocarbons, oxygenated solvents are more polar compounds. Typically, oxygenated solvents are those having hydroxyl or carbonyl groups, and many of them have considerable solubility in water. Other solvents include chlorinated solvents which are fully- or semi-chlorinated hydrocarbons and the rarely used fluorinated hydrocarbons of the Freon types.

Brief Summary Text (8):
This invention is directed to printing screen cleaning or reclaiming compositions which are generally effective in solvating or degrading inks used in the printing industry. The ink cleaning compositions not only solubilize or degrade broad classes of printing inks, but also possess high or no flash points, excellent biodegradabilities and high threshold limit values. Thus, the health and safety of the screen printer or worker in the industry is exceedingly enhanced by this invention. In addition, this invention provides for a system of cleaning and reclaiming compositions which are synergistically operative whereby inks can be removed and the printed emulsion on the screen may be sensitized for effective

removal. In addition, in another of its general aspects, this invention involves a method of cleaning or cleaning and reclaiming printing screens made of silk, textile, metal or other types, without damaging the screen and to place it in an immediate condition for either storage or reuse.

Brief Summary Text (9):
The unique compositions of this invention consist essentially of N-methyl-2-pyrrolidone (herein simply sometimes "NMP"), an oxygenated solvent and a surfactant. It has been found that a non-aqueous system of these essential components will solubilize or degrade a wide variety of polymeric or other inks currently being employed in the modern screen printing industry. This composition has been found to penetrate, emulsify and prevent redeposition of inks during their removal from a variety of common printing screens. It has also been discovered that the NMP, oxygenated solvent and surfactant composition must be non-aqueous in order to effectively clean screens or to sensitize the ink-free screen for subsequent emulsion removal, if desired. It has also been found that an amount of the NMP in the composition may be substituted for by tetrahydrofurfuryl alcohol or certain lower alkylene oxide derivatives thereof and desirable results are achieved at less cost.

Brief Summary Text (10):
In other aspects, a screen cleaning or reclamation process is provided by applying the non-aqueous NMP, oxygenated solvent and surfactant system (herein sometimes simply "NMP Concentrate") onto a screen surface for a sufficient dwell time to enable solubilization or degradation of the ink. Then, the ink may be rinsed with water to remove it from the screen. By this method the ink is degraded to a point whereby a medium to high pressure, low-volume water spray will permit the complete removal of ink. As also disclosed and claimed in the above mentioned copending application entitled "Method of Printing Screen Cleaning and Reclaiming", by Albert B. Cord and Gary M. Valasek, a preferred method for applying the NMP concentrate is to spray it onto the screen ink surface. Spraying a coherent spray of the NMP concentrate enables extremely low amounts to be used and degradation may still surprisingly be achieved. In another feature of the invention after the NMP concentrate is applied, the coated surface of the screen is brushed or mechanically agitated by non-aqueous means to help disintegrate the cleaning composition before water rinsing. It has also been found that, after the ink cleaning step with the NMP concentrate, the emulsion is in a sensitized state for removal from the screen with a periodate-containing emulsion remover. The screen may thus be totally reclaimed. Thereafter, if further desired, any image ink residue or "ghost" as the term is used in the trade, may be removed with a caustic solution containing oxygenated solvent. By the above sequence, this invention also provides for an overall screen cleaning and reclamation process.

Brief Summary Text (11):
As developed in the background of this invention, hot solvent and alkaline techniques have been employed in the prior art screen cleaning and reclamation. This invention avoids the need for such hot cleaning techniques and the associated health hazards created by such techniques. In another of its aspects, the invention is capable of performance at ambient or room temperature conditions. In this essential respect, it is considered highly unexpected and unobvious that a cleaning and reclamation process could operate at such low or ambient temperatures and be as effective in removing a wide variety of ink compositions. Furthermore, whereas it has been disclosed in prior patents to employ derivatives of pyrrolidone including alkyl pyrrolidones, in cleaning compositions, it has not heretofore been suggested that any such pyrrolidone derivative may be employed in a compositions for the removal of screen printing ink compositions. Furthermore, even where pyrrolidones have been suggested in non-analogous arts, they generally are employed in aqueous systems in contrast to the essentially non-aqueous concentrates of this invention. Also, for purposes of this invention, N-methyl-2-pyrrolidone is essentially required to operate in combination with oxygenated solvents and surfactants in order to achieve the most optimum desired results. In substance, in the screen printing industry, it has not been heretofore suggested that a non-toxic, biodegradable and very safe cleaning composition may be provided, and still achieve highly desirable cleaning and synergistic reclamation effects, and has been provided by the compositions of this invention.

Brief Summary Text (13):
As delineated above, the essential components of the inventive compositions are N-methyl-2-pyrrolidone (NMP), an oxygenated solvent and a surfactant. Furthermore, tetrahydrofurfuryl alcohol (THFA) may be substituted for an amount of NMP and desired results are still achieved. Propylene or ethylene oxide adducts of THFA may be used instead of THFA. In a preferred composition, the oxygenated solvents are a combination of butyl cellosolve and cyclohexanone. These preferred oxygenated solvents include esters and ethers, and mixed classes classes thereof. The surfactant is preferable from the group consisting of nonionic or anionic surfactants, or mixtures thereof, and a specific example of nonionic surfactant is octyl phenoxy (polyethoxy) ethanol of Rohm & Haas, sold under the trademark GAFAC RP-710 by General Analine and Film Corporation. Further examples of oxygenated solvents from the class of glycol ethers which may be used include methyl cellosolve, hexyl cellosolve, cellosolve solvent, methyl carbitol, carbitol solvent, butyl carbitol, hexyl carbitol, and the like. Other examples of ketones included methyl ethyl ketone, methyl isobutyl ketone, methyl isoamyl ketone, ethyl butyl ketone, isobutyl keptyl ketone, isophorone, diacetone alcohol, acetone, 4-methoxy-4-methyl-2-pentanone, and the like. Other examples of ethers include butyrolactone, diethyl carbitol and dibutyl carbitol, and others. Examples of esters include butyl lactate, butyl acetate, butyl carbitol acetate, carbitol acetate, butyl cellosolve acetate, cellosolve acetate, 2-ethyl hexyl acetate, amyl acetate, methyl cellosolve acetate, formates, and others. Examples of alcohols include amyl alcohol, butyl alcohol, furfuryl alcohol, 2-butyne-1,4,diol, tetrahydrofurfuryl alcohol, and others. Therefore, in accordance with the broader principles of this invention, oxygenated solvents from the above mentioned classes are suitable for use, depending upon the required solvating capacities of the oxygenated solvents in order to obtain the most desired biodegradability, least flammability and highest threshold limit values to meet or exceed health and safety standards. In accordance with the preferred principles of this invention, however, as mentioned above, there are specific examples which meet all of these criteria in the most preferred aspects of this invention. In a generic aspect, the oxygenated solvent facilitates the low viscosity solvating character of the NMP and helps disperse it to solubilize or degrade the ink compositions. The NMP is also water active when needed. Thus, the combination of the NMP and the oxygenated solvent provide a coaction between organic co-solvents along with a unique water activity to provide a synergistic action in solvating or degrading ink compositions on screens for removal with water. Yet, it is imperative that the NMP concentrates be essentially non-aqueous during solvation or degradation of ink because any significant water will destroy the effectiveness of NMP in its cleaning power as it is used in this invention. However, the solvated or degraded ink must then be in a state for removal with a low-volume, pressurized stream of water.

Brief Summary Text (14):
In addition to the surfactants mentioned above, other nonionic, anionic, cationic and amphoteric surfactants may be used, as listed primarily in McCutcheon's Detergents and Emulsifiers, 1980 Edition, MC Publishing Company, Glenrock, N.J. The surfactants aid in the dispersion and degradation of the inks for aqueous removal. Surfactants of the anionic type may be (1) of the group of saponified fatty acids or soaps, or (2) of saponified petroleum oil such as sodium salts or organic sulfonates or sulfates or (3) of saponified esters, alcohols or glycols, with the letter being well known as anionic synthetic surfactants. Examples of these anionic surfactants include the alkaryl sulfonates or amine salts thereof such as sulfonates of dodecyl benzene or diethanolamine salt of dodecyl benzene sulfonic acid. Most of these sulfonates contain many chemical species. The class name given to most of them is "alkylaryl sulfonate". Simply, this means that a paraffinic hydrocarbon is bonded to an aromatic or benzene nucleus and the aromatic portion has been sulfonated. Examples of saponified fatty acids (C.sub.6 -C.sub.24) are the sodium or potassium salts of myristic, palmitic, stearic, oleic or linoleic acids or mixtures thereof. Also in this class of anionic surfactants are organic phosphate esters including alkali and alkaline earth metal salts of neutral phosphoric acid esters of oxylalkylated higher alkyl phenols or aliphatic monohydric alcohols. "Aerosol OT" is a dioctyl alkali metal sulfosuccinate anionic surfactant made by Cyanamide. The nonionic surfactants suitable for use commonly have hydrophylic portions or side chains usually of the polyoxyalkylene type. The oil soluble or dispersible part of

the molecule is derived from either fatty acids, alcohols, amides or amines. By suitable choice of starting materials and regulating of the length of the polyoxyalkylene chain, the surfactant parts of the nonionic detergents may be varied as is well known. Suitable examples of nonionic surfactants include alkylphenoxy polyoxyethylene glycol, for example, ethylene oxide adduct of either octyl-, nonyl- or tridecyl- phenol and the like. These mentioned nonionic surfactants are usually prepared by the reaction of the alkyl phenol with ethylene oxide. Commercial products are sold under the trademarks "Triton X-100 or X-114" by Rohm and Haas Co. or "Tergitol" by Union Carbide and Carbon Corp. which are alkyl phenyl ethers of polyethylene glycol. Other specific examples of nonionic surfactants include glyceryl monooleate, oleyl monisopropanolamide sorbitol dioleate, alkylol amides prepared by reacting alkanolamides such as monoisopropanolamine, diethanolamine, or monobutanolamine with fatty acids such as oleic, pelargonic, lauric and the like. The cationic surfactants are also well developed and mainly include betaines and quaternary ammonium compounds. Some specific examples of betanies include imidazoline betaines, aliphatic and carboxcyclic betaines, and betaines with hetero atoms in the hydrophobic chains such as dedecyloxypropyldimethyl aminoacetic acid. Typical of the quaternary ammonium compounds that may be mentioned are dimethyl dicoco ammonium chloride, cetyl pyridinuim acetate, methyl cetyl piperidinium proprionate, N,N dilauryl, N,N diemthyl ammonium deithophosphate, and the like. Thus it will be understood that other anionic, cationic, amphoteric or nonionic surfactants may be employed in accordance with the principles of this invention.

Brief Summary Text (15):
The amounts of ingredients vary over wide ranges, however, it is preferred to use a significant amount of the N-methyl-2-pyrrolidone, i.e., about 30-85% by weight. The oxygenated solvent is usually contained in an amount of about 15-35% be weight. The remainder of the composition comprises a surfactant or a mixture of surfactants within the range of about 1 to about 5% by weight. The NMP concentrates may also be supplemented with other organic solvents such as methylene chloride, trichloroethane, dimethyl sulfoxide and its derivatives, fluorocarbons and freons in amounts of about 10-30% by weight where additional faster penetrating power may be desired for better ink solubilization. Such organic solvents are permitted which would not alter the essential characteristics of the ink cleaning compositions of this invention and may help to enhance activity penetrating of the co-solvents in penetrating, emulsifying and accelerating the degradation of the inks for subsequent removal.

Detailed Description Text (3):
75.72% N-methyl-2-pyrrolidone (NMP)

Detailed Description Text (4):
18.52% Butyl Cellosolve

Detailed Description Text (9):
37.86% N-methyl-2-pyrrolidone (NMP)

Detailed Description Text (10):
9.26% Butyl Cellosolve

Detailed Description Text (18):
64.00% N-methyl-2-pyrrolidone (NMP)

Detailed Description Text (23):
0.20% Anionic Surfactant (DUPONT ZONYL FSN)

Detailed Description Text (25):
In this example, THFA is incorporated along with NMP as the primary components of the cleaning composition. It has been found the THFA may be substituted an amount of NMP and the same or similar efficiency is achieved in obtaining the benefits of the invention. Furthermore, cost is reduced even though such properties as low toxicity essentially remain the same in comparison to the use of NMP alone. THFA also has a lower or less noticeable odor. Furthermore, in comparison to the composition of Example 1, this Example 3 composition eliminates the need for butyl cellosolve which is a glycol recently found to have undesirable effects on man and being absorbable

by promoting liver damage. Thus, the employment of a polyalkylene glycol such as diethylene glycol ethyl or methyl ether is more preferred. The UCON trade material used in this example is such a higher glycol. It has also been found the the zonyl anionic surfactant provides better sheeting action or, in other words, in enhances the ability of the cleaning composition to spread out over the screen.

Detailed Description Text (27):
43.00% N-methyl-2-pyrrolidone (NMP)

Detailed Description Text (33):
0.20% Anionic Surfactant (DUPONT ZONYL FSN)

Detailed Description Text (36):
10.00% N-methyl-2-pyrrolidone (NMP)

Detailed Description Text (41):
0.20% Anionic Surfactant (DUPONT ZONYL FSN)

Detailed Description Text (45):
32.00% N-methyl-2-pyrrolidone (NMP)

Detailed Description Text (52):
0.20% Anionic Surfactant (DUPONT ZONYL FSN)

Detailed Description Text (53):
This example is similar in chemical constitution to Example 2 above except that cellosolve acetate and butyl cellosolve are eliminated from the composition with the inclusion of a substantial amount of THFA. Thus, the higher glycols or higher glycol esters are eliminated from this composition and substantial equal amounts of NMP and THFA are employed. It has been found desirable to employ the composition of Example 6 where a faster penetrating capability of carriers is desired for example in connection with inks of the vinyl, epoxy or nylon type.

Detailed Description Text (55):
Before describing in detail the cleaning and reclaiming of printing screens employing the composition of this invention, an understanding of certain underlying facts or terms is important. First of all, the ink side of the printing screen is alternately called the squeegee side, the well side, or the front side, but for purposes of this description it will be call the ink side. The opposite side of the screen is called the back side. The term "ink" will be the generic term for many of the compositions that are employed in the practice of screen printing including dyes or inks commonly referred to as flexible enamels, synthetic enamels, fast-dry enamels, flexible lacquers, industrial lacquers, flat vinyl ink, vinyl half-tone ink, fluorescent vinyl ink, gloss vinyl ink, satin vinyl ink, flock adhesive, transparent ink, metallic powders, acrylic ink, plastisol ink, Mylar ink, textile ink, among many other types of inks. For general information of ink compositions, reference may be had to the catalog by KC Graphics, In. 1978-1979, copyright 1978 by KC Graphics, Inc. Reference may also be had to "Textile Screen Printing" by Albert Kosloff, Second Edition, International Standard Book Number 0-911380-39-6 (1976). These sources will also serve as background information for the inks which may be cleaned with the inventive cleaning compositions. As developed above, the printing screen may be made from a number of materials and may have various mesh sizes. A mono-filament screen is a single strand of material for example of polyester, nylon, stainless steel, silk, chrome-plated wire, or other things, which is woven into a specific number of squares per a dimension, i.e., a 230 mesh means 230 open squares per square inch. A multi-filament screen is comprised of a series of strands of similar materials just mentioned, braided before weaving into the mesh measurement, i.e., 12.times..times.150 mesh would mean 12 interwoven strands subsequently woven into 150 open squares per square inch and, where the cross-over points of mesh occur, pigment can get into the strands and may become extremely hard to remove. Thus, it may be determined by this description of either the mono-filament or the multi-filament screens that the requirements for complete removal of ink residue, for instance, may differ due to the construction of the screen. In particular, that last residue often referred to in the art as "ghost" or "haze" would be more readily apparent in the multi-filament screens where pigment can get into the strands and

becomes very hard to remove. There are a number of classes of emulsions which initially cover the print-contact side of the screen. For instance, a direct emulsion is a water-reducible substance which cures to a temporary, non-removable substance when exposed to specific wavelengths light. For instance, the art work can be placed over the fresh emulsion while it is still reactive to water and exposed to halide light, at which time the part of the emulsion not exposed to the light, because it is covered with the art, strays water-reactive. After a certain shooting or light exposure time, the art is removed, the screen is then flushed with water and the part that has not been exposed to the light is flushed out, leaving the emulsion-void image area through which the ink flows through the screen for printing. The measure of difficulty of emulsion removal also relates to the number of coats which are put on the mesh, that is, dried and reapplied, and so forth, until you have between, for instance, 2 to about 5 coats of emulsion. Thus, the degree of difficulty in removing emulsion depends upon a number of factors including the number of coats, whether it is strongly sensitized, the exposure time, type of light activation and chemical hardeners used after emulsion development, among other factors.

Detailed Description Text (56):
In view of the above background, the cleaning composition of Example 1 is a liquid non-aqueous solvent concentrate having a flash point of approximately 203.degree. F. (95.degree. C.) and it is designed to remove a large variety of inks and paints from screens. In another feature of the invention, NMP concentrate of Example 1 prepares or sensitizes many common emulsions for subsequent removal with a low-volume, high pressure water rinsing. It is fast, efficient, economical by its capability of low-volume usage, biodegradable and does not carry the red label (DOT flammable) solvent designation. It is also safe on all screens and can be left on screens for extended periods prior to rinsing for cleaning. The composition of Example 1 may be employed in a recirculating solvent system or in other portable systems. In the case of a recirculating solvent system, Example 1 concentrate is allowed to flow on the ink side of an inclined screen, whereupon it may be drained for approximately 5 minutes. alternatively, the ink side of the screen may be sprayed in a light even pattern with a cohesive spray to prevent volatilization, followed by a dwell time of about 2-5 minutes. A pressurized unit is operated for example under about 30 to 100 psi (normally 40 psi) to provide a coherent stream of concentrate from a nozzle about 6" to 12" away from the screen. Mists are avoided. In the case of either the recirculating solvent or spray treatment, the degraded inks ar then easily removed by a light to high pressure, i.e., approximately 50 to 100 psi, low-volume water rinse. Low-volume means 2-4 gallons per minute. A fan spray has been found preferred to provide a balance of force and quantity. A suitable fan spray nozzle is the UniJet 65/01 manufactured by Spraying Systems Company of Wheaton, Ill. It is important, as developed above, that the screens on which the ink will be solubilized or degraded are free from water prior to cleaning. The presence of water either in the NMP concentrate or on the screen greatly destroys the effectiveness of the composition.

Detailed Description Text (57):
It has been found that the most preferred cleaning technique for achieving the advantages of the invention is the method disclosed and claimed in said application Serial No. by Cord and Valasek. In said application, the cleaning concentrate is sprayed onto the ink side of the screen in a light even pattern and allowed to dwell for a short period of time, i.e., several minutes up to several hours, depending upon the factors involved in the cleaning system, composition of the inks, production timing, and so forth. During the dwell time, the liquid cleaning concentrate penetrates, emulsifiers and solubilizes the ink. The ink stays on the screen, but its former ink character is destroyed. During the dwell time, the co-solvent actions of N-methyl-2-pyrrolidone and the oxygenated solvent are at work. Furthermore, the surfactant is penetrating the ink composition for dispersion and to aid in later ink removal by water. The coaction of all three ingredients permits the solubilization or degradation of the ink permitting it to be dispersed for removal. The N-methyl-2-pyrrolidone provides water activity to the composition for removal with water, however, water must be under pressurized conditions such that th ink composition may be blown out; i.e., blown away from the screen. Thus, the composition is a delicate balance of ingredients whereby organic components of the ink may be solubilized or degraded by both the N-methyl-2-pyrrolidone and oxygenated

solvents. Furthermore, even though water during the presence of the solubilization and degradation of the ink would be determental to the activity of N-methyl-2-pyrrolidone, nevertheless, the degraded ink in the presence of the NMP and cosolvent is water-active and may be removed from the screen with a pressurized fan of low-volume water rinse. In this connection it is preferred to employ a slicing fan or stream of water and sweeping it across the screen from the bottom upwards in a manner such that the ink by be removed without redeposition.

Detailed Description Text (58):
As indicated above, it is preferred to mechanically agitate the surface of the printing screen after the cleaning concentrate has been sprayed onto the inked surface. Preferably, a dry brush or similarly mechanical means is employed to agitate the surface for the purpose of partially disintegrate the degraded ink composition prior to spraying with water. It has been found that the amount of concentrate employed may be reduced if the mechanical agitation step is added to the method. It is important in this step that the mechanical agitation means be conducted under anhydrous or non-aqueous conditions so that the solvation or disintegration effect continues so that the pressure spray may still be employed to remove the degraded ink. The mechanical action coupled with the chemical action facilitates a better penetrating capability and renders the process more reproducible.

Detailed Description Text (59):
The composition of Example 2 above is employed in the same manner for cleaning as the liquid solvent concentrate of Example 1. However, the presence of methylene chloride tends to enhance the penetration of the entire composition and cellosolve acetate enhances the water solubility of the composition. Other organic solvents which may be substituted for the methylene chloride include other chlorinated solvents like 1,1,1-trichloroethane, dimethyl sulfoxide, its derivatives and fluorocarbons or Freons. In either case, the addition of such an organic solvent which enhances penetration, may also tend to evaporate and, therefore, has a much shorter wet life or dwell time on the screen. For instance, whereas the composition of claim 1 may be left on the screen surface for a number of hours, the composition of Example 2 is usually employed for several minutes, i.e., between 2-5 minutes for example. It is to be further understood that the ink solubilizer of Example 1 may be sequentially used in combination with the cleaning concentrate of Example 2. For instance, during the course of a cleaning operation, the ink may be solubilized with the Example 1 concentrate where an operation may require screens to be left from production for a period of time up to several hours prior to the rinse removal of the ink. In such a case, the dwell time may be followed by a fresh degrading concentrate of Example 2 so that the residue may be activated for subsequent removal with a low-volume, high pressure water rinse. Furthermore, the amounts of the materials sprayed onto the substrate vary but normally they are within the range of several ounces per several square feet, for example, 2-4 ounces per 6 foot square of screen.

Detailed Description Text (60):
Thus, preferably the method involves spraying of the concentrates of either of the Examples 1-6 onto a screen. The spray is an economical and low volume usage followed by dwell time to solubilize or degrade the ink. In another mode, mechanical agitation of the concentrate-sprayed screen surface is employed to help disintegrate the ink. Thereafter, a low-volume, high pressure water stream is directed at the substrate, preferably a fan-spray is employed to slice through and help remove the destroyed ink composition. If the solubilized and degraded screen were simply dipped in water, the screen would not be effectively cleaned. There is a balance between the force and the quantity of the water which is employed which will be understood by the person of ordinary skill in the art in view of this description.

Detailed Description Text (61):
It has been observed in connection with the method of cleaning the screens with a low area coverage spray, that a light mist may tend to settle on other remaining areas of degraded inks. It has been found that this problem can be alleviated or overcome by the addition of another component into the concentrate. That component may be characterized as a hydrophobic additive and in particular it has been found that synthetic water soluble oils sold under the trademark UCON are satisfactory.

Depending upon the amount of water mist or back-lash that is to be expected from the water rise out, part of the surfactant package or liquid NMP concentrate may contain a water dispersible oil which functions initially as a water repellant to a light water spray or mist, but readily allows a low volume, pressurized stream of water to remove the ink compositions previously degraded and solubilized by the NMP concentrate. Such water soluble oils are of the class of polyalkylene glycols, commonly known as UCON lubricants manufactured by Union Carbide Corporation, but other types and mixtures thereof could be used of differing water solubility. A preferred high molecular weight one is UCON 50-HB5100. Specific compounds include polyalkylene glycols, i.e., an oxirane polymer, CAS Registration No. 9038-95-3 or ethoxylated lanolin or ethoxylated castor oil. However, it is preferred that the oils of the types described herein do not leave a residue on the screen after an adequate water rinse.

Detailed Description Text (62):
When the printing screen is to be totally reclaimed, that is, cleaned and the emulsion removed completely, the process is as follows. When the process of printing is completed, the operator cards or squeegees off any residual ink that in on the ink side of the screen as well as any ink from the print-contact side. From a very practical standpoint, inks are expensive and an attempt is made to return as much of the ink as possible. From a standpoint of cleaning, more excess ink requires more cleaning concentrate in order to remove the ink and reclaim the screen without haze or ghost residue. After the screen is well carded, either before or after removal from the screen printing press, the cleaning process begins. If the process is to be an immediate reclaim, either the concentrate of Example 1 or 2 may be used, generally speaking, depending on ink type. Either concentrate is sprayed on the ink side of the screen and then moved to the reclaim area. In the process of immediate reclaiming, within about 5-10 minutes after the application of either of the compositions of Examples 1 or 2, immediate reclaiming should be commenced. After spraying with either concentrate, the image is flushed out from the print-contact side of the screen with high pressure water, as developed above, and this clears all or practically all of the ink. At the same time, the entire surface of the emulsion is wetted with water to prepare it for the application of a periodate-containing emulsion remover. It has been found that there is a synergistic action resulting from use of the non-aqueous concentrate of Example 1 or 2, or for that matter 3 to 6, whose residue remains even after the water spray such that the emulsion is sensitized for faster removal. It is theorized, however, that in the immediate reclaim process, the emulsion is somehow softened or made more permeable for the treatment with the periodate-containing emulsion remover. A suitable example of an emulsion remover is exemplified by Example 7.

Detailed Description Text (64):
94.096% Water

Detailed Description Text (67):
110 grams/1000 pounds batch of an Anionic Surfactant Package of Equal Amounts of GAFAC RP-710, identified above and CALSOFT F-90 (sodium dodecylbenzene sulfonate)

Detailed Description Text (68):
The above periodate containing remover is then sprayed onto the screen surface on the print side and is permitted to dwell there anywhere from about 15 seconds to several minutes. This is generally an adequate time for degradation of the emulsion. Again a high pressure spray is employed to clean the emulsion from the screen. As developed above, it is important that residual effects of the concentrate of Examples 1 to 6 after the water spray be utilized immediately by following with an application of the periodate-containing emulsion remover. It has been proven that when more time elapses, for instance one-half hour, the emulsion will be taken off with greater difficulty. Accordingly, there is a synergism between the residual effect of the cleaning compositions of this invention insofar as they coact with such periodate containing emulsion removers and provide the complete removal of the emulsion.

Detailed Description Text (69):
Finally, in the event that there is a residual ghost image as explained above, especially in connection with a multi-filament screen, a ghost or image remover may

be employed. It is to be noted, however, that although there are at some time residual images, they are not necessarily a hindrance to further use of the screen and some screen printers are not particular where there is a tinge of residue as long as the mesh squares themselves are not blocked in the screen. If a ghost or haze remover is used, it is typically a caustic solution of oxygenated solvents. Methylene chloride is also added to the caustic solution. The reason for such usage is that they are water rinsable or soluble and leave no residue to cause emulsion problems later. In the case of the ghost or haze removers of this type it is necessary to brush, roll or card them on as opposed to spraying because of the enhanced viscosity. A typical composition is as follows:

Detailed Description Text (76):
For screens which will be delayed in processing but which will be totally reclaimed, it is important to destroy the ink so that at some time thereafter, there is in a matter of hours, the screen may be treated. For instance, the composition of Example 1 is sprayed onto the ink side and, again, this is a cohesive spray of the type referred to above without mist so as to reduce volatilization. The screen is thus wet and the ink tends to stay wet for a period of time. However, if there is a lapse of time before it goes to the screen shop for complete removal of the emulsion, there will be some tendency for drying and possibly some run-down on the print side of the screen. If this develops, then the print side of the screen may be resprayed with a composition of Example 2 which acts to freshen up the solubilized residue after treatment with the composition of Example 1 to degrade the ink prior to being contacted with the water spray. At this point, upon respraying the print-contact side of the screen with a concentrate of Example 2, and rinsing the emulsion is sensitized or conditioned for the subsequent action with the emulsion cleaner as mentioned above. Again, if a ghost or haze occurs, the ghost remover may be employed as set forth above.

CLAIMS:

1. A printing screen ink cleaning composition for removing inks from images on said screen and for performance at room temperature consisting essentially of a non-aqueous biodegradable liquid mixture of about 30 to 85% N-methyl-2-pryyolidone, about 10 to 35% of an oxygenated solvent and about 1-5% of a surfactant.

2. The composition of claim 1 wherein about 30 to 64% tetrahydrofurfuryl alcohol is substituted for an amount of said N-methyl-2-pyrrolidone or said oxygenated solvent.

3. The composition of claim 1 wherein the oxygenated solvent is selected from the group consisting of a butyl cellosolve, cellosolve acetate and cylcohexanone, and mixtures thereof.

4. The composition of claim 3 wherein said surfactant is selected from the class consisting of nonionic, anionic and amphoteric surfactants and mixtures thereof.

6. The composition of claim 1 which additionally includes a water dispersible oil in an amount which functions in said composition initially as a repellant to a light water spray but allows a stream of water to remove said inks from said screen.

7. The composition of claim 6 wherein the water dispersible oil is selected from the group consisting of polyalkylene glycol, ethoxylated lanolin and ethoxylated castor oil.

8. A method of cleaning a printing screen having a residue of ink on the surface thereof which comprises the steps of treating at room temperature the printing screen ink residue with a non-aqueous biodegradable liquid composition consisting essentially of about 30 to 85% of N-methyl-2-pyrrolidone, about 10 to 35% oxygenated solvent and about 1-5% surfactant,

allowing the composition to dwell on the ink surface for a sufficient period of time to degrade the ink residue, and

rinsing the degraded ink residue with a pressurized stream of water.

10. The method of claim 8 wherein rinse water is applied as a fan spray.

11. The method of claim 8 wherein water rinse is a pressurized low-volume water rinse.

12. The method of claim 8 wherein the oxygenated solvent is selected from the group consisting of butyl cellosolve, cellosolve acetate and cyclohexanone, and mixtures thereof.

14. The method of claim 13 wherein after several hours dwell time the degrded ink is treated with a liquid concentration of about 30 to 85% of N-methyl-2-pyrrolidone, about 10 to 35% oxygenated solvent, methylene chloride, and about 1-5% surfactant prior to rinsing.

15. The method of claim 8 wherein said liquid composition additionally includes a water dispersible oil in an amount which functions in said composition initially as a repellant to a light water spray but allows a stream of water to remove said inks from said screen.

16. The method of claim 15 wherein said water dispersible oil is selected from the group consisting of polyalkylene glycol, ethoxylated lanolin and ethoxylated castor oil.

17. A method of cleaning and reclaiming a printing screen having a printed image of an emulsion and a residue of ink on the surface thereof which comprises the steps of

treating at room temperature the printing screen ink residue with a non-aqueous biodegradable liquid composition consisting essentially of about 30 to 85% N-methyl-2-pyrrolidone, about 10 to 35% oxygenated solvent and about 1-5% surfactant,

allowing the composition to dwell on the ink surface for a sufficient period of time to degrade the ink residue,

rinsing the degraded ink residue with a pressurized stream of water,

contacting within a short time the water-rinsed screen with a periodate-containing emulsion remover, and

flushing the resultant screen with a stream of pressurized water to remove the emulsion image.

19. The method of claim 18 wherein said periodate-containing emulsion remover additionally contains an anionic surfactant.

20. The method of claim 17 wherein after the last water flush a caustic solution of oxygenated solvent is applied to the screen to remove ghost images.

21. The method of claim 20 wherein the caustic solution of oxygenated solvent comprises a solution of sodium hydroxide, a oxygenated solvent from the group consisting of cyclohexanone, cellosolve acetate and mixtures thereof and one other surfactant.

23. The method of claim 21 wherein said other surfactant is an anionic surfactant.

24. The method of claim 17 wherein, after said dwell time and prior to the first water ringse, spraying onto the resultant screen residue a composition comprising about 30 to 85% N-methyl-2-pyrrolidone, about 10 to 35% oxygenated solvent and about 1-5% surfactant, and about 10-30% organic solvent from the group consisting of methylene chloride, 1,1,1-trichloroethane, dimethylsulfoxide and trichlorotrifluoroethane, and mixtures thereof.

**End of Result Set**



L12: Entry 90 of 90 File: DWPI May 12, 1987

DERWENT-ACC-NO: 1987-150202
DERWENT-WEEK: 198721


TITLE: Compsn. for removing ink from printing screen - contg. N-methyl-2-pyrrolidone, oxygenated solvent and surfactant

Basic Abstract Text (1):
Printing screen ink cleaning compsn. comprises a non-aq., biodegradable liq. mixt. of 30-85% N-methyl-2-pyrrolidone (NMP), 10-35% oxygenated solvent (I) and 1-5% surfactant.

Basic Abstract Text (2):
Opt. 30-64 wt.% tetrahydrofurfuryl alcohol replaces some of the NMP or (I). (I) is butyl cellosolve, cellosolve acetate and/or cyclohexanone, and (II) is nonionic, anionic and/or amphoteris.

Basic Abstract Text (3):
USE/ADVANTAGE - The compsn. is used to remove ink from images on printing screesn. It degrades and solubilises a wide range of ink residues for quick removal of ink with a pressurised water stream, and is active at room temp. It meets health and safety requirements as regards biodegradability, lack of flammability and threshold limit values.

Standard Title Terms (1):
COMPOSITION REMOVE INK PRINT SCREEN CONTAIN N METHYL PYRROLIDONE OXYGENATE SOLVENT SURFACTANT

WEST

**End of Result Set**

Generate Collection Print

L12: Entry 90 of 90 File: DWPI May 12, 1987

DERWENT-ACC-NO: 1987-150202
DERWENT-WEEK: 198721


TITLE: Compsn. for removing ink from printing screen - contg. N-methyl-2-pyrrolidone, oxygenated solvent and surfactant

INVENTOR: VALASEK, G M

PATENT-ASSIGNEE: INTERCONTINENTAL CHEM CO LTD (ITCC)

PRIORITY-DATA: 1984US-0619067 (June 11, 1984), 1981US-0327782 (December 7, 1981), 1985US-0807123 (December 11, 1985)

PATENT-FAMILY:

| PUB-NO | PUB-DATE | LANGUAGE | PAGES | MAIN-IPC |
|---|---|---|---|---|
| US 4664721 A | May 12, 1987 | | 009 | |

APPLICATION-DATA:

| PUB-NO | APPL-DATE | APPL-NO | DESCRIPTOR |
|---|---|---|---|
| US 4664721A | December 11, 1985 | 1985US-0807123 | |

INT-CL (IPC): B08B 7/00; B08B 30/00; C11D 1/18

ABSTRACTED-PUB-NO: US 4664721A
BASIC-ABSTRACT:

Printing screen ink cleaning compsn. comprises a non-aq., biodegradable liq. mixt. of 30-85% N-methyl-2-pyrrolidone (NMP), 10-35% oxygenated solvent (I) and 1-5% surfactant.

Opt. 30-64 wt.% tetrahydrofurfuryl alcohol replaces some of the NMP or (I). (I) is butyl cellosolve, cellosolve acetate and/or cyclohexanone, and (II) is nonionic, anionic and/or amphoteris.

USE/ADVANTAGE - The compsn. is used to remove ink from images on printing screesn. It degrades and solubilises a wide range of ink residues for quick removal of ink with a pressurised water stream, and is active at room temp. It meets health and safety requirements as regards biodegradability, lack of flammability and threshold limit values.

ABSTRACTED-PUB-NO: US 4664721A
EQUIVALENT-ABSTRACTS:

CHOSEN-DRAWING: Dwg.0/0

DERWENT-CLASS: A97 E13 G02 P43
CPI-CODES: A08-S02; A11-C07; A12-W07F; A12-W11; E07-D03; G02-A03C; G04-B08; G05-F;